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1 Jul 94

## APPENDIX I

### CHECKLISTS

#### FOR

#### ON-SITE LABORATORY INSPECTIONS

## CHECKLISTS FOR ON-SITE LABORATORY INSPECTION

**CHARTS I-1** through **I-36** contain checklists on which the adequacy of laboratory organization, facility, equipment, operation, and QA/QC policy and practice shall be checked by the inspector(s) during an on-site inspection. The titles of these checklists are:

CHART I-1	Organization and Personnel
CHART I-2	Facilities
CHART I-3	Equipment
CHART I-4	General QA/QC
CHART I-5	Report Generation
CHART I-6	Field Sampling
CHART I-7	Sample Receipt and Storage
CHART I-8	Sample Preparation for Organic Analysis
CHART I-9	General QA/QC for Organic Analysis by GC
CHART I-10	Organic Analysis by GC: HVO (8010A)
CHART I-11	Organic Analysis by GC: TPH (Modified 8015)
CHART I-12	Organic Analysis by GC: AVO (8020)
CHART I-13	Organic Analysis by GC: PHENOLS (8040A)
CHART I-14	Organic Analysis by GC: PEST/PCB (8080)
CHART I-15	Organic Analysis by GC: PAH (8100)
CHART I-16	Organic Analysis by GC: HERB (8150A)
CHART I-17	General QA/QC for Organic Analysis by GC/MS
CHART I-18	Organic Analysis by GC/MS: VOA (8240A)
CHART I-19	Organic Analysis by GC/MS: BNA (8270A)
CHART I-20	Organic Analysis by GC/MS: DIOXINS (8280)
CHART I-21	Organic Analysis by HPLC: PAH (8310)
CHART I-22	Organic Analysis by HPLC: EXPLOSIVES (8330)
CHART I-23	Sample Preparation for Metal Analysis
CHART I-24	General QA/QC for Metal Analysis
CHART I-25	Metal Analysis by ICP: METALS (6010A)
CHART I-26	Metal Analysis by AA: METALS (7000s)
CHART I-27	General QA/QC for Classical Analysis
CHART I-28	Classical Analysis: COMMON ANIONS (300s)
CHART I-29	Classical Analysis: OIL AND GREASE (413.1)
CHART I-30	Classical Analysis: TRPH (418.1)
CHART I-31	Classical Analysis: CYANIDE (9010A)
CHART I-32	Classical Analysis: TOC (9060)
CHART I-33	Waste Characteristics: Ignitability (1010/1020)
CHART I-34	Waste Characteristics: Corrosivity (1110)
CHART I-35	Waste Characteristics: Reactivity (Section 7.3)
CHART I-36	Waste Characteristics: Toxicity (1311)

An inspector(s) should use those checklists that are applicable to the laboratory to be inspected. Depending on the projects and/or the laboratory, an inspector(s) shall determine which sections of each checklist should be used. The inspector(s) should check those items, under the "YES" column, which he/she believes to be adequately practiced and documented in the laboratory. Additional information should be entered in the "COMMENT" columns with an "N/A" for items not applicable to the work for the USACE. The detail of any observations, comments, or problems should be recorded in the blank space provided at the end of each checklist. Any deficiencies noted on the checklist shall be discussed with and acknowledged by the laboratory management staff during an Exit Interview.

The checklists will be revised or augmented when revised or new analytical methods are officially approved by the USEPA or other regulatory agencies. All revisions of the checklists shall be approved by the HQUSACE.

**CHART I-1**

**ORGANIZATION AND PERSONNEL:**

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ITEM	YES	COMMENT
Is the lab legally identifiable?		
Has the lab provided supervision by persons familiar with the test methods, the objective of the test, and the assessment of the results?		
Has the lab specified and documented the responsibility, authority, and relationship of all personnel who manage, perform, or verify work affecting the quality of tests?		
Does the lab have a QA Officer who has responsibility for the quality system and its implementation?		
Is the QA Officer familiar with all test procedures and QC requirements?		
Does the QA Officer have direct access to the highest level of management at which decisions are taken on lab policy or resources?		
Does the lab nominate deputies in case of absence of the QA Officer?		
Does the lab have documented protocol for training in QC methods?		
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of tests?		
Is each analyst accountable for performing tasks in any of the following areas meet the specified minimum experience:  a. Inorganic sample preparation - 6 months?  b. Organic sample preparation - 1 year?		

CHART I-1

ORGANIZATION AND PERSONNEL:

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ITEM	YES	COMMENT
c. Classical analysis - 1 year? d. Trace metal analysis - 1 year? e. Gas chromatography - 1 year? f. Pesticide residue analysis - 2 years? g. Mass spectrometry - 1 year? h. Spectrum interpretation - 2 years? i. Radiochemical analysis - 2 years?		
Is each analyst's performance audited and approved prior to work without close supervision by a senior chemist?		
Is there documented evidence of analyst proficiency for each test method performed?		
Does the lab have an in-house training program or send staff to training schools?		
Are staff's qualification, training, and experience recorded?		
Is backup provided for technical staff?		
Does the lab have documented policy and procedures to ensure the protection of clients' confidential information and proprietary right?		

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**CHART I-1**

**ORGANIZATION AND PERSONNEL:**

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ITEM
Additional observations, comments, or problems:

**CHART I-2**

**FACILITIES:**

Page 1 of 3

ITEM	YES	COMMENT
Does the lab building have a security system?		
Is access to the test and sample storage area controlled?		
Is a guest logbook available and used?		
Is equipment protected and environment monitored as needed?		
Does the lab have adequate work space, ventilation, light, and access to stable power sources at workstations?		
Is the lab clean and organized?		
Is the lab free of dust, drifts, and temperature extremes?		
Is reagent water free of contamination used for preparation of standards and blanks?		
Is the conductivity of water routinely checked and recorded on a daily basis?		
Is a separate conductivity meter (capable of being calibrated) used to measure the conductivity of the reagent water? (Meters built into the water purification system are not acceptable.)		
Is a corrective action taken when the conductivity of the reagent water is two micromho or greater at 25°C?		
Are exhaust hoods provided to allow contamination-free work with volatile and hazardous materials?		

**CHART I-2**

**FACILITIES:**

Page 2 of 3

ITEM	YES	COMMENT
Is the air flow of the hoods periodically checked and recorded?		
Are adequate facilities, including cold storage, provided for separate storage of samples, extracts, reagents, solvents, reference materials and standards to preserve their identity, concentration, purity, and stability?		
Is adequate chemical storage space available and are chemicals properly segregated according to class?		
Are solvent storage cabinets properly vented as appropriate for the prevention of possible lab contamination?		
Does the lab have adequate safety devices such as eye wash stations, spill control stations, showers, first-aid stations, etc.?		
Are these safety devices checked routinely to ensure they are still working properly?		
Are special facilities (e.g., glove box, controlled air) provided for handling extremely toxic materials such as dioxin?		
Are adequate filing space available for storage of manuals, SOPs, raw data, and reports?		
Are chemical waste disposal policies and procedures well-defined and followed by the lab?		

**CHART I-2**

**FACILITIES:**

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ITEM
Additional observations, comments, or problems:

CHART I-3

EQUIPMENT:

Page 1 of 8

ITEM	YES	COMMENT
Is appropriate equipment available for use in accordance with required methodology?		
Is equipment adequately maintained with sufficient spare parts and are maintenance instructions available?		
Is out-of-service equipment clearly labelled?		
Are equipment maintenance logs maintained?		
Are standard curves prepared to cover the expected concentration ranges of samples?		
Are calibration logs maintained?		
Is a new curve prepared annually (or more frequently if specified by the method) or whenever new reagents are prepared, whichever is more frequent?		
Are calibration labels used as applicable?		
Is proper backup equipment available?		
Balances:  a. Analytical Balances:  (1) Are analytical balances capable of weighing 0.1 mg in use?  (2) Is there a record of balance calibration in two ranges with Class S weights? (Please specify the ranges.)  (3) Do records show daily functional and calibration checks ( $\pm 0.1\%$ ) for analytical balances?		

CHART I-3

EQUIPMENT:

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ITEM	YES	COMMENT
<p>(4) Have the balances been calibrated at least annually?</p> <p>b. Top Loading and/or Pan Balances:</p> <p>(1) Is a top loading and/or pan balances capable of accurately detecting a 100 mg weight at a load of 150 g available?</p> <p>(2) Is there a record of the balance having been serviced within the previous 12 months?</p> <p>(3) Is there a record of balance calibration in two ranges with Class S weights? (Please specify the ranges.)</p> <p>(4) Do records show weekly functional and calibration checks (<math>\leq \pm 0.1\%</math>) for pan balances?</p> <p>(5) Have the balances been calibrated at least annually?</p>		
<p>Thermometers:</p> <p>a. Certified Thermometer:</p> <p>(1) Does the lab have, or have access to, an NIST-traceable factory certified thermometer?</p> <p>(2) Is a copy of factory certificate for the thermometer available for inspection?</p> <p>(3) Is there a record of the annual check of the certified thermometer at the ice point?</p>		

CHART I-3

EQUIPMENT:

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ITEM	YES	COMMENT
<p>b. Working Thermometers:</p> <p>(1) Are sufficient working thermometers available so that each has a dedicated use?</p> <p>(2) Does each working thermometer have a unique identifying number?</p> <p>(3) Is the calibration of each working mercury thermometer checked annually against an NIST-traceable thermometer?</p> <p>(4) Is the calibration of each dial type thermometer checked at least quarterly against an NIST-traceable thermometer?</p> <p>(5) Are digital thermometers calibrated quarterly at their temperature of use against an NIST-traceable thermometer?</p> <p>(6) Is a record of thermometer calibration maintained?</p>		
<p>pH Meters:</p> <p>a. Is a clean pH meter with appropriate electrode with scale graduations at least 0.1 pH units in use?</p> <p>b. Is a thermometer or temperature sensor for automatic compensation in use?</p> <p>c. Do records show daily, or before each use, calibration, whichever is less frequent?</p> <p>d. Are three standard buffers used for the calibration?</p>		

**CHART I-3**

**EQUIPMENT:**

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ITEM	YES	COMMENT
<p>e. Are aliquots of standards of pH 4, pH 7, and pH 10 used only once?</p> <p>f. Are acceptance limits in place?</p> <p>g. Is the meter recalibrated if not within the limits of 0.05 for two point calibration and within 0.2 for one point calibration?</p> <p>h. If the limits cannot be achieved, is the problem determined and resolved?</p>		
<p>Conductivity Meters:</p> <p>a. Are a conductivity meter and probe of sufficient sensitivity in use?</p> <p>b. Do records show a daily, or before each use, calibration check, whichever is less frequent?</p> <p>c. Do records show cell constant is determined annually?</p> <p>d. If the cell constant has a large deviation from the expected value, is the cause determined and corrected?</p>		
<p>Refrigerators/Walk-in Coolers:</p> <p>a. Is a thermometer in each refrigerator with bulb immersed in liquid?</p> <p>b. Are thermometers graduated in increments no larger than 1°C?</p> <p>c. Are temperatures for each refrigerator recorded daily?</p>		

**CHART I-3**

**EQUIPMENT:**

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ITEM	YES	COMMENT
d. Do records show that refrigerator temperatures are maintained in the range of 2 to 6°C?		
<p>Ovens:</p> <p>a. Are thermometers graduated in increments no larger than 1°C?</p> <p>b. If the oven temperature cannot be read without opening the door, is the bulb of the thermometer in a sand bath?</p> <p>c. Is oven temperature adequately monitored (e.g., beginning and end of each use cycle)?</p> <p>d. Is a record documenting date, time of use, nature of use, and temperature maintained?</p> <p>e. Do the records indicate the oven holds temperature at the appropriate drying temperature?</p>		
<p>Glassware:</p> <p>a. Is the lab stocked with sufficient volumetric glassware for the analyses performed?</p> <p>b. Is Class A volumetric glassware available for standard preparation?</p> <p>c. Is glassware cleaned in a manner appropriate for the analytical procedures for which it is to be used?</p> <p>d. Is glassware cleaning procedure posted next to the cleaning station?</p>		

CHART I-3

EQUIPMENT:

Page 6 of 8

ITEM	YES	COMMENT
<p>e. For organics, are the following basic cleaning steps used?</p> <p>(1) Removal of surface residuals immediately after use?</p> <p>(2) Flush with methanol before it is placed in hot detergent soak?</p> <p>(3) Hot soak (&gt;50°C) in a synthetic detergent bath to loosen and float most particulate material?</p> <p>(3) Hot-water rinse to flush away floated particulates?</p> <p>(4) Soak with oxidizing agent such as chromic acid solution made up of sulfuric acid and potassium or sodium bichromate at 40-50°C to destroy traces of organic compounds?</p> <p>(5) Hot-water rinse to flush away materials loosened by the deep penetrant soak?</p> <p>(6) Distilled-water rinse to remove metallic deposits from the tap water?</p> <p>(7) Methanol or isopropanol rinse to flush off any final traces of organic materials and remove the water?</p> <p>(8) Flushing the item immediately before use with some of the same solvent that will be used in the analysis?</p>		

CHART I-3

EQUIPMENT:

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ITEM	YES	COMMENT
f. Is glassware for organics dried at 100°C?		
g. As an alternative to solvent rinsing, is glassware for organics heated to a minimum of 300°C to vaporize any organics?		
h. Is volumetric glassware, glassware with ground glass joints, or sintered glassware not heated to high temperature to avoid deformation?		
i. For trace metals, is the plastic or glassware cleaned with detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and reagent water?		
j. Is chromic acid not used to clean glassware and plastic bottles for trace metal analysis?		
k. Is clean glassware properly covered and stored to prevent recontamination by dust?		

**CHART I-3**

**EQUIPMENT:**

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ITEM
Additional observations, comments, or problems:

**CHART I-4**

**GENERAL QA/QC:**

Page 1 of 10

ITEM	YES	COMMENT
Does the lab maintain a QA Manual?		
<p>Does the manual address the important elements of a QA/QC program, including the following:</p> <ul style="list-style-type: none"> <li>a. QA Policy and Objectives?</li> <li>b. organization?</li> <li>c. Personnel?</li> <li>d. Facilities and Equipment?</li> <li>e. Document Control?</li> <li>f. Sample Receiving and Storage?</li> <li>g. Analytical Methodology?</li> <li>h. Instrument Operation?</li> <li>i. Instrument Calibration?</li> <li>j. Preventive Maintenance?</li> <li>k. Certification of Regents/Standards?</li> <li>l. Data Generation/Reduction/Validation?</li> <li>m. Data Reliability?</li> <li>n. Feedback and Corrective Action?</li> <li>o. Recordkeeping and Archives?</li> <li>p. Internal QC Audits?</li> <li>q. Performance and External Audits?</li> <li>r. Training/Certification of Personnel?</li> </ul>		

CHART I-4

GENERAL QA/QC:

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ITEM	YES	COMMENT
Is the manual available to all laboratory personnel?		
Is the manual updated regularly?		
Is line authority for all referenced organizations explained by including an organization chart?		
Is the organizational structure appropriate to accomplish the project QA objectives?		
Are QA/QC responsibilities and reporting relationships clearly defined?		
Are all staff aware of QA/QC and its application?		
Is the QA Officer a full-time employee?		
Does the QA Officer operate independently of the analyses?		
Does the QA Officer report directly to a senior officer?		
Are internal QA reviews conducted at least annually and recorded including any corrective action taken?		
QA Objectives and Criteria:  a. Are the terms and definitions for precision, accuracy, comparability, representativeness, and completeness properly used?  b. Have the following been defined for each parameter and matrix:  (1) Level of QA effort (frequency and type of QC, etc.)?		

CHART I-4

GENERAL QA/QC:

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ITEM	YES	COMMENT
(2) Accuracy (matrix spikes, surrogate spikes, reference samples, etc.)? (3) Precision (replicate samples)? (4) Sensitivity or MDL? (5) Statistical reporting units? c. Are quantitative limits established for each parameter and matrix? d. Are field and lab both covered? e. If appropriate, are completeness objectives quantitatively stated? f. Are representativeness and comparability appropriately addressed?		
Is a sample batch clearly defined and determined as a group of samples of $\leq 20$ , with similar matrix, prepared and analyzed with same technique and reagents at same time or within same time sequence?		
Is at least the following minimum QC practiced in the lab? a. For Inorganic/Classical Analysis: (1) Minimum three concentrations of standards plus blank, and one check standard in ten; the lab shall repeat all samples if check standard is outside $\pm 10\%$ . (2) One method blank per batch. (3) One matrix spike per batch. (4) One lab duplicate per batch.		

CHART I-4

GENERAL QA/QC:

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ITEM	YES	COMMENT
<p>(5) One control (consists of a control matrix spiked with analytes representative of the target analytes) per batch.</p> <p>b. For Organic Analysis:</p> <p>(1) Minimum five concentrations of standards plus blank and one check standard in ten; if any are outside control limits repeat all samples.</p> <p>(2) One method blank per batch.</p> <p>(3) One matrix spike per batch.</p> <p>(4) One lab duplicate/matrix spike duplicate per batch.</p> <p>(5) One control (consists of a control matrix spiked with analytes representative of the target analytes) per batch.</p> <p>(6) Surrogates for all samples.</p>		
Are there any exceptions to the above minimum QC practice in the lab?		
Has the lab established control limits for all the above types of QC samples? (Control limits should be at least as tight as those stated in the methods.)		
Are quality control data (e.g., standard curve, results of duplicates and spikes) accessible for all analytical results?		
Are method detection limits empirically determined and documented?		

**CHART I-4**

**GENERAL QA/QC:**

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ITEM	YES	COMMENT
Are control charts maintained for each routine analysis?		
Do lab records indicate what corrective action has been taken when results fail to meet QC criteria?		
Are documented methods/procedures available for assurance of field and lab equipment functioning optionally?		
Is a program of initial and periodic calibration established for each method?		
Does the QA Manual include calibration documentation requirements: a. Date of calibration? b. Identification of standards used? c. Personnel performing calibration? d. Results of calibration (raw data and summary statistics)? e. Corrective actions taken?		
Are primary reference standards used for calibration only?		
Are all working standards versus primary standards verified and documented?		
Are reference materials traceable to NIST standards?		
Are reagent grade or higher purity chemicals used to prepare standards?		

**CHART I-4**

**GENERAL QA/QC:**

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ITEM	YES	COMMENT
Are fresh analytical standards prepared at a frequency consistent with good QC?		
Are reference materials/reagents properly labeled with concentrations, dates of preparation and expiration, and identity of the person preparing the reagent?		
Are updated equipment operating instructions available?		
Are analytical procedures written as SOPs available for review?		
Are all procedural steps and options described?		
Are the criteria of method selection included (e.g., in order to obtain a specific data quality objective?)		
If method choice is governed by regulatory requirement (e.g., NPDES, SDWA, RCRA), have the appropriate methods been chosen?		
Are approved methods being used as specified?		
Are procedures documented for data handling, reporting, and recordkeeping?		
Are documented validation procedures applied at appropriate levels for all measurement procedures?		
Are documented procedures available for checking the validity of reported analysis values?		

**CHART I-4**

**GENERAL QA/QC:**

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ITEM	YES	COMMENT
Are predetermined limits available for data acceptability beyond which corrective action is required?		
Are documented procedures available for correcting erroneously reported results?		
<p>Are the following SOPs available for review?</p> <ul style="list-style-type: none"> <li>a. Sample collection, preservation, storage, and handling.</li> <li>b. Sample preparation and analysis.</li> <li>c. Purity and preparation of standards.</li> <li>d. Instrument operation and calibration.</li> <li>e. Preventive maintenance and corrective actions.</li> <li>f. Quality control for each type of test.</li> <li>g. Quality control chart.</li> <li>h. Data reduction and reporting.</li> <li>i. Recordkeeping and archives.</li> <li>j. Personnel training/certification.</li> <li>k. Procurement and inventory procedures.</li> <li>l. Glassware cleaning.</li> <li>m. Waste disposal.</li> <li>n. Technical and managerial review of lab operation.</li> </ul>		

CHART I-4

GENERAL QA/QC:

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ITEM	YES	COMMENT
Are procedures in place for making and controlling revisions to in-house SOPs?		
Are there internal audits for both field and lab activities?		
Are there designated persons who will conduct the audits? Auditor's Name: _____ _____ _____		
Is there a documented protocol which will be used for audit?		
Are acceptance criteria defined?		
Are audit reports prepared and distributed? To whom? _____ _____ _____		
Are the type and frequency of audit reports specified? Type/frequency: _____ _____ _____		
Do the reports address:  a. status of project (time table)?  b. results of performance and system audits?  c. data quality assessment?  d. significant QA problems and proposed corrective action?		

CHART 1-4

GENERAL QA/QC:

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ITEM	YES	COMMENT
Does the lab participate in any external proficiency testing programs such as EPA performance evaluation studies for water supply and water pollution?		
Are there corrective actions taken and documented?		
Does the lab have a laboratory information management system (LIMS) currently in use?		
If yes, manufacturer: _____		
Model No.: _____		
Brief description of hardware & software:		
Does the LIMS have an audit trail feature?		

CHART I-4

GENERAL QA/QC:

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ITEM
Additional observations, comments, or problems:

**CHART I-5**

**REPORT GENERATION:**

Page 1 of 4

ITEM	YES	COMMENT
Are there documented procedures for internal field and lab checks of:  a. precision and accuracy?  b. routine duplicates, spikes, and standard samples?  c. statistical methods, including control chart and computer methods?		
Is there a written description of the lab record system including data management, review, validation, and audit?		
Is there written description of the lab reporting system?		
Is there a system in place that provides for retrievability and traceability of the sample source, analytical methods, results, person performing analysis, and date?		
Are records and reports adequately secured for the required amount of time to ensure the integrity per regulatory requirements?		
Are permanently bound notebooks with consecutively numbered pages being used?		
Is a unique serial number clearly displayed on each notebook cover or spine?		
Are logbook entries made in permanent fashion with indelible ink?		
Are logbook entries legible?		
Are all raw data signed and dated by the chemist who performed the analysis?		

**CHART I-5**

**REPORT GENERATION:**

Page 2 of 4

ITEM	YES	COMMENT
Are there evidence of entries being tampered with?		
Has data been altered?		
If yes, was a single line drawn through the entry and corrections made without obliterating original entries?		
Was the new entry initialed and dated?		
Were technical reviews conducted on all logbook entries and deliverables?		
Was a minimum of three-levels of technical reviews conducted by chemist, supervisor, QA Officer?		
Have QC measures been utilized to ensure the quality of the work performed?		
Can all signatures be clearly identified?		
Is a central file being maintained for all project documents?		
Is a system of document control numbers in place?		
Are all completed lab notebooks, raw data, analytical reports, electronic tapes and disks, and other pertinent documentation filed in a secure, controlled archives area?		
Has the supervisor personally examined and reviewed each notebook periodically and signed and dated the review?		
Do the lab's reports accurately, clearly, and unambiguously present results and all other relevant information?		

**CHART I-5**

**REPORT GENERATION:**

Page 3 of 4

ITEM	YES	COMMENT
<p>Does each test report include the following information:</p> <ul style="list-style-type: none"> <li>a. Names and addresses of laboratory and client?</li> <li>b. Unique identification and page number?</li> <li>c. Case narrative?</li> <li>d. Sample identification and description?</li> <li>e. Dates of sample receipt and test performed, as appropriate?</li> <li>f. Identification of sample preparation and analysis methods used?</li> <li>g. Description of any deviations from test method?</li> <li>h. Disclosure of any subcontractor used?</li> <li>i. Results including all method required QC data?</li> <li>j. Description of any problems or failures identified?</li> <li>k. Measurement uncertainty, if relevant?</li> </ul>		
Is the lab's report format acceptable?		
Does the length of storage time for all sample related information comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)		

**CHART I-5**

**REPORT GENERATION:**

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ITEM
Additional observations, comments, or problems:

CHART I-6

**FIELD SAMPLING:** (Complete if this lab conducts field sampling for USACE projects.)

Page 1 of 3

ITEM	YES	COMMENT
Is a site specific Chemical Data Acquisition Plan (CDAP) available to lab personnel?		
Are lab personnel familiar with the QC requirements of the CDAP?		
Do sampling procedures follow contract specifications?		
Do field documentation procedures: <ul style="list-style-type: none"> <li>a. document the sources and lot numbers of reagents and supplies?</li> <li>b. include procedures/forms for recording the exact location and specific considerations associated with sample acquisition?</li> <li>c. document specific preservative methods?</li> <li>d. include labels containing all necessary information?</li> <li>e. include forms for tracking custody?</li> </ul>		
Is there a unique identification on each sample?		
Is sampling information properly recorded such as sample ID numbers, type (grab versus composite), preservatives, analytes, location, date and time of collection, and name of sample collector?		
Are written chain-of-custody procedures available for review? Are they in accordance with USACE/EPA guidelines?		

**CHART I-6**

**FIELD SAMPLING:**

Page 2 of 3

ITEM	YES	COMMENT
Are there written sampling SOPs covering sampling plan, sampling equipment, sample collection, preservation, identification, storage, and lab handling?		
Are there written descriptions of chain-of-custody of samples? (Attach a copy of chain-of-custody form.)		
Are there written procedures for field measurement of flow, dissolved oxygen, residual chlorine, etc.?		
Are there written procedures for monitoring water supply, effluent, ambient air, stacks, radiation, etc.?		
Are proper preservation techniques being used for the analytical methods and sample types concerned?		
Are provisions made for the collection of QA/QC split samples?		
Are provisions made for field blanks and duplicate samples at an appropriate rate (normally 10% or minimum of one per matrix type, whichever is greater, or as specified in contract?)		
Are adequate facilities available to do compatibility testing?		

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**CHART I-6**

**FIELD SAMPLING:**

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ITEM
Additional observations, comments, or problems:

**CHART I-7**

**SAMPLE RECEIPT AND STORAGE:**

Page 1 of 3

ITEM	YES	COMMENT
Are there adequate written procedures for receipt and storage of samples to ensure sample integrity?		
Do the written procedures address sample handling, storage, and dispersment for analysis and disposal?		
Do the written procedures accurately reflect procedures in use?		
Is separate area and facility including hoods available for sample receipt?		
Is a dedicated sample custodian available? Custodian's name: _____		
Are appropriate chain-of-custody procedures documented and followed in the lab?		
Does the lab maintain internal custody procedures?		
Does a permanent record exist for sample log-in?		
Are samples assigned unambiguous sample ID numbers when logged in?		
Is a checklist used to document problems or deficiencies noted during sample log-in?		
Is sample temperature properly measured and recorded during log-in?		
Are pH values of aqueous samples for the following analyses checked and adjusted in a hood during log-in? (Metals, phenols, oil and grease, TRPH, TOC, TOX, COD, hardness, ammonia, nitrate-nitrite, total phosphorus, Kjeldahl and organic nitrogen, radiological testing, cyanide, and sulfide.)		

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**CHART I-7**

**SAMPLE RECEIPT AND STORAGE:**

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ITEM	YES	COMMENT
Is the pH value properly measured to avoid sample contamination and to minimize waste generation?		
Are corrective actions properly documented?		
Are clients notified if problems are noted?		
Are there adequate facilities for sample storage?		
Are samples stored in such a way as to maintain their identity, integrity, stability, and concentration?		
Are volatile organic samples stored in separate refrigerators from other samples?		
Are temperature logs of storage coolers and refrigerators properly maintained?		
Are acceptable temperature ranges used and posted? ( $4 \pm 2^{\circ}\text{C}$ )		
Are coolers and refrigerators locked when unattended?		
Is final disposition of samples documented?		

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**CHART I-7**

**SAMPLE RECEIPT AND STORAGE:**

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ITEM
Additional observations, comments, or problems:

CHART I-8

**SAMPLE PREPARATION FOR ORGANIC ANALYSIS:**

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for sample preparation?</li> <li>b. Do these SOPs accurately reflect procedures in use?</li> <li>c. Are all sample preparations conducted in a hood?</li> <li>d. Are a group of samples (up to a maximum of 20) which behave similarly with respect to the procedures being employed and which are processed as a unit with the same method sequence and the same lots of reagents and with the reagents and with the manipulations manipulations common to each samples within the same time period or in continuous sequential time periods considered as a batch?</li> <li>e. Are the following lab internal QC samples prepared for each batch of samples? <ul style="list-style-type: none"> <li>(1) Method blanks?</li> <li>(2) Matrix spikes?</li> <li>(3) Matrix spike duplicates?</li> <li>(4) Matrix duplicates?</li> <li>(5) Laboratory control samples?</li> </ul> </li> <li>f. If the quantity of field samples is not sufficient for internal QC analyses, are blank spike/blank spike duplicate or duplicate laboratory control samples analyzed?</li> </ul>		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
g. Is a purified solid matrix used for preparation of method blanks for soil and sediment volatile organics?		
h. Is a purified sodium sulfate used for preparation of method blanks for soil and sediment semivolatile organics including pesticides, herbicides, and PCBs?		
i. If sample extracts are cleaned up with Methods 3600s, are the associated QC samples also processed through the corresponding cleanup methods?		
j. Is the water meniscus of aqueous samples marked on the side of sample container for later determination of sample volume?		
k. Are the rates of internal QC samples consistent with method requirements or, at a minimum, 5% per batch of no more than 20 samples with similar matrix, whichever is greater?		
l. Is the appropriateness of a particular preparation for a specific sample type determined by the completeness of extraction and by spike recoveries?		
m. Are logbooks for sample preparation used and well maintained?		
n. Are permanently bound notebooks with consecutively numbered pages used?		
o. Is a unique serial number clearly displayed on each notebook?		
p. Are critical times entered in logbooks?		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
<p>q. Are spiking solutions traceable to NIST or other reliable standards?</p> <p>r. Are spiking solutions labeled properly with date of preparation, composition, concentration and identity of preparer?</p> <p>s. Have entries been made in permanent fashion and corrections made without obliterating original entries?</p> <p>t. Are corrections reviewed and initialed by a supervisor?</p> <p>u. Does the logbook of sample preparation contain the following information?</p> <p>(1) Date/time?</p> <p>(2) Sample ID number?</p> <p>(3) Sample preparer?</p> <p>(4) Matrix noted?</p> <p>(5) Spiking standards?</p> <p>(6) Pretreatment?</p> <p>(7) Volume/weight of sample?</p> <p>(8) Final volume?</p> <p>(9) Preparation methods?</p>		
<p>Separatory Funnel Liquid-Liquid Extraction Method 3510A):</p> <p>a. Is the following equipment available?</p>		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
<p>(1) Separator funnel (2-L with Teflon stopcock)?</p> <p>(2) Drying tube with Pyrex glass wool at bottom and a Teflon stopcock?</p> <p>(3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?</p> <p>(4) Water bath capable of temperature control within 5°C?</p> <p>b. Are enough sets of separator funnels (2,000 mL with Teflon stopcock) and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?</p> <p>c. Are the following reagents available?</p> <p>(1) Sodium hydroxide solution (10 N)?</p> <p>(2) Sulfuric acid solution (1:1)?</p> <p>(3) Anhydrous sodium sulfate?</p> <p>(4) Methylene chloride?</p> <p>(5) Hexane?</p> <p>(6) 2-Propanol?</p> <p>(7) Cyclohexane?</p> <p>(8) Acetonitrile?</p> <p>d. Are surrogate standards and spiking solutions added to the samples in the separator funnel prior to the addition of methylene chloride?</p>		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
<p>e. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C?</p> <p>f. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately, and refrigerated?</p>		
<p>Continuous Liquid-Liquid Extraction (Method 3520A):</p> <p>a. Is the following equipment available?</p> <p>(1) Continuous liquid-liquid extractor equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication?</p> <p>(2) Drying column with Pyrex glass wool at bottom and a Teflon stopcock?</p> <p>(3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?</p> <p>(4) Water bath capable of temperature control within 5°C?</p> <p>(5) Heating mantle (Rheostat controlled)?</p> <p>b. Are enough sets of continuous liquid-liquid extractors and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?</p> <p>c. Are the following reagents available?</p> <p>(1) Sodium hydroxide solution (10 N)?</p>		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
(2) Sulfuric acid solution (1:1)? (3) Anhydrous sodium sulfate? (4) Methylene chloride? (5) Hexane? (6) 2-Propanol? (7) Cyclohexane? (8) Acetonitrile? d. Are surrogate standards and spiking solutions added to the samples prior to extraction? e. Is twice the volume of spiking solution added when GPC cleanup will be used? f. Are samples extracted for 18-24 hours at a specific pH value? g. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C? h. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
Soxhlet Extraction (Method 3540A): a. Is the following equipment available? (1) Soxhlet extractor with 500-mL round bottom flask?		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
<p>(2) Drying column with Pyrex glass wool at bottom and a Teflon stopcock?</p> <p>(3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?</p> <p>(4) Heating mantle (Rheostat controlled)?</p> <p>(5) Grinding apparatus?</p> <p>b. Are enough sets of Soxhlet extractors and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?</p> <p>c. Are the following reagents available?</p> <p>(1) Anhydrous sodium sulfate?</p> <p>(2) Toluene/Methanol (10:1) solvent?</p> <p>(3) Acetone/Hexane (1:1) solvent?</p> <p>(4) Methylene chloride?</p> <p>(5) Hexane?</p> <p>(6) 2-Propanol?</p> <p>(7) Cyclohexane?</p> <p>(8) Acetonitrile?</p> <p>d. If a dry waste sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, is it processed into a homogeneous sample that meet these requirements?</p>		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
e. Are surrogate standards and spiking solutions added to the samples prior to extraction?  f. Is twice the volume of spiking solution added when GPC cleanup will be used?  g. Are samples extracted for 16-24 hours?  h. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C?  i. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
Sonication Extraction (Method 3550):  a. Is the following equipment available?  (1) Grinding apparatus?  (2) Horn-type sonicator equipped with a titanium tip (475 W)?  (3) Sets of Kuderna-Danish glassware (including concentration tubes, evaporation flasks, and macro and micro Snyder columns)?  (4) Drying column with Pyrex glass wool at bottom and a Teflon stopcock?  (5) Water bath capable of temperature control within 5°C?  b. Are the following reagents available?  (1) Anhydrous sodium sulfate?		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
(2) Methylene chloride/Acetone (1:1)?		
(3) Methylene chloride?		
(4) Hexane?		
(5) 2-Propanol?		
(6) Cyclohexane?		
(7) Acetonitrile?		
c. If the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, is it processed into a homogeneous sample that meet these requirements?		
d. Are samples mixed with anhydrous sodium sulfate to form a free flowing powder?		
e. Are surrogate standards and spiking solutions added to the samples prior to the addition of the extraction solvent?		
f. Is twice the volume of spiking solution added when GPC cleanup will be used?		
g. Are samples that are expected to contain low concentrations of organics sonicated three times for three minutes with fresh solvent each time?		
h. Are samples that are expected to contain high concentrations of organics sonicated once for two minutes?		
i. Is the Kuderna-Danish concentration process conducted with a hot water bath at 80-90°C?		

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SAMPLE PREPARATION FOR ORGANIC ANALYSIS:

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ITEM	YES	COMMENT
j. If concentrated extracts are to be stored more than two days are they transferred to Teflon-lined screw-cap or crimp-top vials, labeled appropriately and refrigerated?		
<p>Purge-and-Trap (Method 5030A):</p> <p>a. Is a purge-and-trap device available?</p> <p>b. Are purge-and-trap systems subjected to a periodic bake-out and cleaning process and are these actions documented?</p> <p>c. Is a purge chamber designed to accept 5-mL samples available?</p> <p>d. Is a 25-mL all glass purge chamber available for GC/MS Methods 524.1, 524.2, and 8260 (optional)?</p> <p>e. Is the gaseous headspace less than 15 mL?</p> <p>f. Is the trap a minimum of 25-cm long?</p> <p>g. For Method 8010A, is the trap packed with 1.0-cm 3% OV-1 on Chromosorb-W 60/80 mesh, 7.7-cm Tenax GC, 7.7-cm silica gel, and 7.7-cm charcoal or equivalent?</p> <p>h. For Method 8015A, is the trap packed with 1.0-cm 3% OV-1, 15-cm Tenax GC, and 7.7-cm silica gel or equivalent?</p> <p>i. For Methods 8020 and 8030A, is the trap packed with 1.0-cm 3% OV-1 on Chromosorb-W and 23-cm Tenax GC or equivalent?</p>		

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**SAMPLE PREPARATION FOR ORGANIC ANALYSIS:**

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ITEM	YES	COMMENT
<p>j. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments?</p> <p>k. Is the methanol purge-and-trap quality or equivalent?</p> <p>l. Are surrogate standards and spiking solutions added to the purging chamber along with the sample?</p> <p>m. Is a method blank carried through all of sample preparation and measurement before any sample are processed?</p>		
<p>Headspace (Method 3810):</p> <p>a. Is this method only used as a screening procedure?</p> <p>b. Is a hot bath capable of maintaining a 90°C temperature available?</p> <p>c. Are 125-mL hype-vials with seals and septa used for the equilibration?</p> <p>d. Are both a 1-ppm spike and a 1-ppm standard analyzed along with samples?</p> <p>e. Are the vials with the samples (the 1-ppm spike and the 1-ppm standard) equilibrated in a 90°C water bath for one hour?</p> <p>f. Are the vials maintained at 90°C while 2-mL of headspace gas is withdrawn for direct injection into a GC?</p> <p>g. Is the GC operated using the same GC conditions listed in the method being screened (8010A, 8015A, 8020, 8030A, or 8040A)?</p>		

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**SAMPLE PREPARATION FOR ORGANIC ANALYSIS:**

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Additional observations, comments, or problems:

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GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC:

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ITEM	YES	COMMENT
Are the following detectors available and are they used appropriately?  a. Flame Ionization (603, 604, 609, 610, 8015A, 8030A, 8040A, 8060, 8090, 8100)?  b. Photoionization (602, 8020)?  c. Electron Capture (604, 606, 608, 609, 612, 8040A, 8060, 8080, 8090, 8120, 8150A)?  d. Electrolytic Conductivity (601, 611, 8010A, 8080, 8140)?  e. Microcoulometric (601, 611, 8140)?  f. Thermal Energy Analyzer (607)?  g. Nitrogen/Phosphorus (607, 8140)?  h. Flame Photometric (8140)?		
Are the column ovens, at a minimum, capable of temperature control within $\pm 0.2^{\circ}\text{C}$ at $220^{\circ}\text{C}$ ?		
Are the injection ports glass lined?		
Are manufacturer's operating manuals readily available to bench chemists?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Have any instruments been modified in any way?		
Are the instruments properly vented?		

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GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC:

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ITEM	YES	COMMENT
Is glassware for organics solvent rinsed or heated to a minimum of 300°C to vaporize any organics in a muffle furnace after careful cleaning?		
Is this high temperature treatment avoided for volumetric glassware, glassware with ground joints, or sintered glassware?		
Is there a calibration protocol available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Is a 5-point calibration used?		
Is the calibration curve or calibration factor verified each working day?		
Are calibration results kept in a permanent logbook?		
Is the MDL for each analyte and matrix type determined every six months or whenever there is a significant change in background or instrument response?		
Is the linear calibration range determined for each analyte when there is significant change in instrument response and every six months for those analytes that periodically approach their linear limits?		
Is a method blank included with each sample batch and carried through the entire preparation and analysis?		
Is a matrix spike and a matrix spike duplicate run with each batch at a rate of 5% or one per batch, whichever is greater?		

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**CHART I-9**

**GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC:**

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ITEM	YES	COMMENT
Is corrective action taken if matrix spike recoveries exceed QC limits?		
Is a matrix duplicate run with each batch at a rate of 5% or one per batch, whichever is greater?		
Is corrective action taken if percent differences based on duplicated analyses exceed QC limits?		
Are surrogate recoveries run on each sample?		
Is corrective action taken if surrogate recoveries exceed QC limits?		
Is an LCS prepared with standards independent of calibration standards analyzed for each batch of samples?		
Is corrective action taken if the LCS recovery exceeds QC limits?		
Are QC data statistically analyzed and charted for quality control?		
Are control charts maintained and readily available to bench chemists?		

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GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC:

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ITEM
Additional observations, comments, or problems:

CHART I-10

ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for HVO sample preparation/analysis?</li> <li>b. Do these SOPs accurately reflect procedures in use?</li> <li>c. Are all target analytes, at a minimum, that have retention times published in Table 1 of Method 8010A, routinely analyzed at the lab?</li> <li>d. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>e. Are prenumbered, bound notebooks used for data entry?</li> <li>f. Are all records written in indelible ink?</li> <li>g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible?</li> <li>h. Are corrections initialed and dated by the responsible individual?</li> <li>i. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms?</li> <li>b. Are backup bench chemists available?</li> </ul>		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Does the lab have adequate air handling system to avoid cross contamination of samples?		
c. Is a temperature-programmable gas chromatography equipped with an purge-and-trap device and electrolytic conductivity detector available?		
d. Is oven temperature stable to $\pm 0.5^{\circ}\text{C}$ or better at desired setting?		
e. Is one of the following GC column available?		
(1) 8-ft x 0.1-in ID SS or glass column packed with 1% SP-1000 on Carbowack-B 60/80 mesh or equivalent?		
(2) 6-ft x 0.1-in ID SS or glass column packed with chemically bonded n-octane on Porasil-C 100/200 mesh or equivalent?		
f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		
g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
h. Is helium used as carrier gas? i. Is a hood available for sample preparation? j. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available? k. Are backup instruments available?		
Reagents: a. Is reagent water used free from interferents at the MDL of target analytes? b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available? c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol? d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw-cap at -10 to -20°C and protected from light? e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a problem? f. Are stock standards for target analytes of low boiling points (<30°C) and high reactivity prepared fresh every two months or sooner?		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>g. Are secondary standards stored with minimal headspace and check frequently for degradation or evaporation?</p> <p>h. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to 24 hours)?</p> <p>i. Is a 25-μL Hamilton 702N microsyringe or equivalent used for standard preparation? (Pipets should never be used to dilute or transfer volatile samples or aqueous standards.)</p> <p>j. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards?</p> <p>k. Is "purge-and trap", "pesticide quality" or equivalent methanol stored away from other solvents?</p> <p>l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples?</p> <p>b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?</p>		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?</p> <p>d. Is one of the calibration standards at a concentration near, but above, the MDL?</p> <p>e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?</p> <p>f. Is a linear calibration curve with a correlation coefficient <math>\geq 0.995</math> prepared for each analyte?</p> <p>g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working ranges?</p> <p>h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?</p> <p>i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than <math>\pm 15\%</math> or exceeds the acceptance criteria listed in the Table 3 of Method 8010A?</p>		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?</p> <p>k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?</p>		
<p>Sample Preparation:</p> <p>a. Is a combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane used as surrogate standards?</p> <p>b. Are samples routinely introduced into the GC using purge-and-trap (Method 5030)?</p> <p>c. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments?</p> <p>d. Is direct injection used only for water soluble compounds that do not purge or when concentrations are expected to exceed 10,000 µg/L?</p> <p>e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Is daily calibration checked with a mid-concentration standard at the beginning and the end of an analysis sequence?</p>		

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ITEM	YES	COMMENT
b. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds $\pm 15\%$ when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, that contain target analytes that exceed the criteria?		
c. Are daily retention time windows established for each analyte prior to sample analysis?		
d. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
e. Is the same sample introduction method used for calibration standards and samples? (i.e., either purge-and-trap or direct injection, but not mixed methods.)		
f. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample that has been properly sealed and stored prior to use?		
g. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
h. Is a second GC column used to resolve the analytes from co-eluting non-target compounds?		
i. Are positive hits routinely confirmed by a second GC column?		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>Quality Control:</p> <ul style="list-style-type: none"> <li>a. Are all QC data maintained and available for easy reference and inspection?</li> <li>b. Is a three-level data review carried out within the lab prior to data release?</li> <li>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</li> <li>d. Is the lab specific MDL equal to or lower than the method specified MDL?</li> <li>e. Is a mid-concentration standard analyzed for each group of ten samples in the analysis sequence?</li> <li>f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is greater?</li> <li>g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? <ul style="list-style-type: none"> <li>(1) Is an LCS, prepared with standards independent of calibration standards, analyzed for each batch?</li> <li>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</li> </ul> </li> </ul>		

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ITEM	YES	COMMENT
<p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever is higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever is higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul>		

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ORGANIC ANALYSIS BY GC: HVO (8010A)

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ITEM	YES	COMMENT
<p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>i. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates that encompass the method specified temperature range?</p> <p>j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?</p> <p>k. Are control limits for each surrogate in a given matrix calculated based on the above data?</p> <p>l. Do the control limits fall within the control limits of Method 8240 if applicable?</p> <p>m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p>		

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**ORGANIC ANALYSIS BY GC: HVO (8010A)**

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ITEM	YES	COMMENT
<p>n. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?</p> <p>o. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>p. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p>		

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ITEM	YES	COMMENT
b. Are lab wastes disposed of properly such that no secondary pollution is generated from sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation:  a. Does the lab have sound technical capability for HVO analysis?  b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for HVO analysis?		

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**ORGANIC ANALYSIS BY GC: HVO (8010A)**

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ITEM
Additional observations, comments, or problems:

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for TPH sample preparation and analysis as gasoline range organics (GRO) and diesel range organics (DRO)?</li> <li>b. Do these SOPs accurately reflect procedures in use?</li> <li>c. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>d. Are prenumbered, bound notebooks used for data entry?</li> <li>e. Are all records written in indelible ink?</li> <li>f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</li> <li>g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms?</li> <li>b. Are backup bench chemists available?</li> <li>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</li> </ul>		

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ITEM	YES	COMMENT
<p>Apparatus and Facilities:</p> <ul style="list-style-type: none"> <li>a. Is working space adequate and clean?</li> <li>b. Does the lab have adequate air handling system to avoid cross contamination of samples?</li> <li>c. Is a temperature-programmable gas chromatography equipped with a purge-and-trap device and flame ionization detector available for GRO?</li> <li>d. Is oven temperature stable to <math>\pm 0.5^{\circ}\text{C}</math> or better at desired setting?</li> <li>e. Is a data system available for determination of peak area sums using forced baseline and baseline projection?</li> <li>f. Are the following GC columns available?</li> </ul> <p>GRO Analysis:</p> <ul style="list-style-type: none"> <li>(1) 105-m x 0.53-mm ID Restek RTX 502.2 0.3-micron film thickness?</li> <li>(2) Other capillary columns which can resolve 2-methylpentane from the methanol solvent front in a 25 <math>\mu\text{g/L}</math> LCS and to resolve ethylbenzene from m/p-xylene (&lt;25% valley)?</li> </ul> <p>DRO Analysis:</p> <ul style="list-style-type: none"> <li>(1) 25-m x 0.25-mm Quadrex 007 5% methyl phenyl 0.5-micron film thickness?</li> <li>(2) 30-m x 0.53-mm ID Quadrex RTX-5, 1.5-micron film thickness?</li> </ul>		

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
<p>(3) Other capillary columns which can resolve C<sub>17</sub>/pristane and C<sub>18</sub>/phytane (&gt;50% resolution)?</p> <p>g. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</p> <p>h. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>i. Has any instrument been modified in any way?</p> <p>j. Is a hood available for sample preparation?</p> <p>k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>l. Is a horn-type sonicator equipped with a titanium tip and 475 Watt available in the lab?</p> <p>m. Are backup apparatus available?</p>		
<p>Reagents:</p> <p>a. Is reagent water used free from interferences at the MDL of target analytes?</p> <p>b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?</p> <p>c. Are "pesticide quality" or equivalent solvents used for TPH analysis?</p>		

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
d. Is granular, anhydrous sodium sulfate purified by heating at 400°C for four hours prior to use?		
e. Does the lab use a calibration standard composed of a blend of the following typical ten gasoline compounds for GRO?		
2-methylpentane	15% wt.	
2,2,4-trimethylpentane	15%	
heptane	5%	
benzene	5%	
toluene	15%	
ethylbenzene	5%	
m-xylene	10%	
p-xylene	10%	
o-xylene	10%	
1,2,4-trimethylbenzene	10%	
f. Does the lab use a calibration standard composed of a blend of the following typical 14 C <sub>10</sub> -C <sub>28</sub> even normal alkane standards, plus n-C <sub>17</sub> , pristane, and phytane for DRO?		
decane	≈7% wt.	
dodecane	≈7%	
tetradecane	≈7%	
hexadecane	≈7%	
heptadecane	≈7%	
pristane	≈7%	
octadecane	≈7%	
phytane	≈7%	
eicosane	≈7%	
docosane	≈7%	
tetracosane	≈7%	
hexacosane	≈7%	
octacosane	≈7%	
5-α-androstane (I.S.)	≈7%	

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ITEM	YES	COMMENT
g. Are the materials of interest, if available, or the same type of petroleum fraction, if it is known and original sample is unavailable, used for preparation of calibration standards?		
h. Is an internal standard used for DRO samples to correct for injection variances and matrix interferences?		
i. Does the lab have, at a minimum, the following Pattern Recognition Standards for identification of petroleum hydrocarbons? Gasoline, aviation fuel, (JP-4), kerosene, and diesel fuel (#2).		
j. Does the lab use a well characterized gasoline (e.g., API PS-6 or equivalent) and a commercial diesel #2 as LCSs for GRO and DRO, respectively?		
k. Are stock standards for GRO prepared in methanol and replaced after six months, or sooner, if comparison with check standards indicates a problem?		
l. Are stock standards for DRO prepared in acetone and replaced after six months, or sooner, if comparison with check standards indicates a problem?		
m. Are secondary dilution standards in methanol stored with minimum headspace for volatiles and frequently checked for signs of degradation/evaporation?		
n. Are working standards at a minimum of five concentration levels prepared in reagent water?		

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ITEM	YES	COMMENT
o. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:  a. Are triplicate water samples in 40 mL VOA vials received for GRO analysis?  b. Are water GRO samples inverted and checked for existence of air bubbles?  c. Are soil GRO samples in wide mouth glass jars with Teflon-lined septa checked (without opening the container) for existence of excess headspace?  d. Are duplicate samples collected for the alternate methanol extraction method?  e. Are low level GRO samples stored at 4°C in a separate refrigerators from high level GRO samples?  f. Are GRO samples analyzed within 14 days from collection?  g. Are water DRO samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?  h. Are soil DRO samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		

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ITEM	YES	COMMENT
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Is the GC system calibrated with a minimum of five concentration levels of calibration standard blenders?</p> <p>d. Is one of the calibration standards at a concentration near, but above, the MDL?</p> <p>e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?</p> <p>f. Are the calibration standards for GRO injected using a purge-and-trap?</p> <p>g. For DRO, is a methylene chloride blank run in every batch to determine the area generated on normal baseline bleed between C<sub>10</sub> and C<sub>28</sub> and subtracted from the total areas of DRO standards and samples?</p> <p>h. Is a linear calibration curve with a correlation coefficient <math>\geq 0.995</math> prepared for each analyte?</p> <p>i. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working ranges?</p>		

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ITEM	YES	COMMENT
<p>j. Is the calibration curve or factor verified with, at a minimum, a midpoint calibration standard at the beginning and end of each analysis sequence?</p> <p>k. Is a new calibration curve prepared for any target analyte when the response factors for the daily calibrations vary from the initial response factors by more than 15%?</p> <p>l. Is the retention time window established with three injections of each calibration standard over the course of a 72-hour period?</p> <p>m. Are the retention time windows, specially for surrogates, internal standards, and the first and the last components in calibration standards, checked on a quarterly basis or whenever a new GC column is installed?</p> <p>n. Are the retention times for surrogates, internal standards, and the first and the last components in the daily mid-concentration standard used as the midpoints of the windows for that day?</p>		
<p>Sample Preparation:</p> <p>a. Is a purge-and-trap device used to inject water GRO samples to a GC?</p> <p>b. Are soil and solid GRO samples extracted by methanol extraction, diluted in water and injected with purge-and-trap to a GC?</p> <p>c. Are all supernatant liquids retained in methanol extraction process for GRO samples?</p>		

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ITEM	YES	COMMENT
<p>d. Are water DRO samples extracted by EPA Methods 3510/3520 and soil DRO samples by EPA Methods 3540/3550?</p> <p>e. Is the water meniscus on the side of bottle marked for later determination of the sample volume?</p> <p>f. Is the pH of water DRO samples checked and adjusted with 10 N NaOH or 1:1 H<sub>2</sub>SO<sub>4</sub> to 5-9?</p> <p>g. Is the water DRO sample containers rinsed with methylene chloride? (Do not cap and shake the bottle.)</p> <p>h. Is continuous extraction method used if emulsion forms and cannot be broken during separatory funnel method such that the recovery of methylene chloride is less than 80% after correction for water volubility of methylene chloride?</p> <p>i. For soil DRO samples, are large rocks or foreign materials removed and any vegetation chopped into small pieces?</p> <p>j. Are soil DRO samples sonicated for 1.5 minutes at 475 watts, one second pulse mode with a 50% duty cycle?</p> <p>k. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Are the concentrations of all analytes within the initial calibration ranges?</p>		

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
b. Is a method/instrument blank analyzed after a sample that produces a saturated response from a compound?		
c. If the method/instrument blank is not free from interferences, is the system decontaminated before sample analysis?		
d. Is the quantitation of GRO based on the area summation of all peaks that are above instrument blank baseline and elute between 2-methylpentane and 1,2,4-trimethylbenzene?		
e. Are non-petroleum hydrocarbons, such as chlorinated solvents, ketones, and esters, excluded from the GRO quantitation?		
f. Is the total peak area for C <sub>10</sub> -C <sub>28</sub> from baseline-to-baseline used for DRO quantitation?		
g. Are non-petroleum hydrocarbons, such as chlorinated solvents, phenols, and phthalates, excluded from the DRO quantitation?		
h. Are retention times and patterns of the peaks used in identification of the type of petroleum hydrocarbons?		
i. Are comments provided for contaminants that appear in the GRO and DRO windows but do not match the reference fuels?		
j. Is internal standard, 5 - $\alpha$ -androstande, used as a retention time marker for DRO samples?		

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab before data release?</p> <p>c. Are lab specific MDL or PQL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific PQL equal to or lower than the method specified PQL?</p> <p style="padding-left: 40px;"><b>GRO:</b> water 100 µg/L soil 5 mg/kg</p> <p style="padding-left: 40px;"><b>DRO:</b> water 100 µg/L (diesel #2) soil 4 mg/kg (diesel #2)</p> <p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. For GRO/GRO samples, are duplicate LCSs analyzed at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>g. Is the percent recovery of LCS larger than 50% and the percent difference less than 20%?</p> <p>h. Is a column bleed profile run for each batch of DRO samples to determine the area from normal baseline bleeding and subtracted from the area of DRO samples?</p>		

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ITEM	YES	COMMENT
<p>i. Is a methylene chloride blank run after samples of highly concentrated to prevent carryover?</p> <p>j. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p>		

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ORGANIC ANALYSIS BY GC TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
<p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>k. Does the lab use one or two surrogate compounds, p-chlorofluorobenzene, bromofluorobenzene, or trifluorotoluene, to monitor the system performance and effectiveness of the of the GRO method in dealing with each matrix?</p> <p>l. Does the lab use one or two surrogate compounds, n-pentacosane (n-C<sub>25</sub>) or ortho-terphenyl, to monitor the system performance and effectiveness of the of the DRO method in dealing with each matrix?</p> <p>m. Has the lab established control limits for surrogate recoveries?</p> <p>n. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?</p> <p>o. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>p. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for TPH analysis?</p>		

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ORGANIC ANALYSIS BY GC: TPH (MODIFIED 8015)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for TPH analysis?		
Additional observations, comments, or problems:		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
General:  a. Are written SOPs available and adequate for AVO sample preparation/analysis?  b. Do these SOPs accurately reflect procedures in use?  c. Are manufacturer's operating manuals readily available to bench chemists?  d. Are prenumbered, bound notebooks used for data entry?  e. Are all records written in indelible ink?  f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible?  g. Are corrections initialed and dated by the responsible individual?  h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff:  a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC system and in interpretation of chromatograms?  b. Are backup bench chemists available?  c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

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ITEM	YES	COMMENT
<p>Apparatus and Facilities:</p> <p>a. Is working space adequate and clean?</p> <p>b. Does the lab have adequate air handling system to avoid cross contamination of samples?</p> <p>c. Is a temperature-programmable gas chromatography equipped with an purge-and-trap device and photo-ionization detector available?</p> <p>d. Is oven temperature stable to <math>\pm 0.5^{\circ}\text{C}</math> or better at desired setting?</p> <p>e. Is one of the following GC column available?</p> <p>(1) 6-ft x 0.082-in ID SS or glass column packed with 5% SP-1000 and 1.75% Bentone-34 on 100/120 mesh Supelcoport or equivalent?</p> <p>(2) 8-ft x 0.1-in ID SS or glass column packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on 60/80 mesh Chromosorb W-AW or equivalent?</p> <p>(3) Is column one used as the primary analytical column and column two as a confirmation column?</p> <p>f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</p> <p>g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p>		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
<p>h. Has any instrument been modified in any way?</p> <p>i. Is a hood available for sample preparation?</p> <p>j. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>k. Are backup instruments available?</p>		
<p>Reagents:</p> <p>a. Is reagent water used free from interferences at the MDL of target analytes?</p> <p>b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?</p> <p>c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol?</p> <p>d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw-cap at -4°C and protected from light?</p> <p>e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a problem?</p> <p>f. Are secondary standards stored with minimal headspace and checked frequently for degradation or evaporation?</p>		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
<p>g. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to 24 hours)?</p> <p>h. Is a 25 µL Hamilton 702N microsyringe or equivalent used for standard preparation? (Pipets should never be used to dilute or transfer volatile samples or aqueous standards.)</p> <p>i. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards?</p> <p>j. Is "purge-and-trap", "pesticide quality", or equivalent methanol stored away from other solvents?</p> <p>k. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples?</p> <p>b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p>		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
<p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?</p> <p>d. Is one of the calibration standards at a concentration near, but above, the MDL?</p> <p>e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?</p> <p>f. Is a linear calibration curve with a correlation coefficient <math>\geq 0.995</math> prepared for each analyte?</p> <p>g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?</p> <p>h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?</p> <p>i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than <math>\pm 15\%</math> or exceeds the acceptance criteria listed in the Table 3 of Method 8020?</p> <p>j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?</p>		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
<p>Sample Preparation:</p> <p>a. Are surrogate compounds, bromochlorobenzene, bromofluorobenzene, 1,1,1-trifluorotoluene, fluorobenzene, and difluorobenzene, which encompass the temperature range of this method used for all samples?</p> <p>b. Are samples routinely introduced into the GC using purge-and-trap (Method 5030)?</p> <p>c. Is methanolic extraction of purge-and-trap only used for medium-concentration soils or sediments?</p> <p>d. Is direct injection used only for water soluble compounds that do not purge or when concentrations are expected to exceed 10,000 µg/L?</p> <p>e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Is daily calibration performed with a mid-concentration standard at the beginning and the end of an analysis sequence?</p>		

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ITEM	YES	COMMENT
<p>b. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds <math>\pm 15\%</math> when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria?</p> <p>c. Are daily retention time windows established for each analyte prior to sample analysis?</p> <p>d. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?</p> <p>e. Is the same sample introduction method used for calibration standards and samples? (i.e., either purge-and-trap or direct injection, but not mixed methods.)</p> <p>f. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample that has been properly sealed and stored prior to use?</p> <p>g. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?</p> <p>h. Is a second GC column used to resolve the analytes from co-eluting non-target compounds?</p> <p>i. Are positive hits routinely confirmed by a second GC column?</p>		

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ITEM	YES	COMMENT
<p>Quality Control:</p> <ul style="list-style-type: none"> <li>a. Are all QC data maintained and available for easy reference and inspection?</li> <li>b. Is a three-level data review carried out within the lab prior to data release?</li> <li>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</li> <li>d. Is the lab specific MDL equal to or lower than the method specified MDL?</li> <li>e. Is a mid-concentration standard analyzed for each group of 10 samples in the analysis sequence?</li> <li>f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is greater?</li> <li>g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations? <ul style="list-style-type: none"> <li>(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?</li> <li>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</li> </ul> </li> </ul>		

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ITEM	YES	COMMENT
<p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <p>- the regulatory limit, if any; or</p>		

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ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
<p>- the larger of either five times the expected background or LCS concentrations?</p> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>i. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?</p> <p>j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, once a minimum of 30 samples of the same matrix have been analyzed?</p> <p>k. Are control limits for each surrogate in a given matrix calculated based on the above data?</p> <p>l. Do the control limits fall within the control limits of Method 8240 if applicable?</p>		

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ITEM	YES	COMMENT
<p>m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>n. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?</p> <p>o. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>p. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		

CHART I-12

ORGANIC ANALYSIS BY GC: AVO (8020)

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ITEM	YES	COMMENT
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for AVO analysis?</p> <p>b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____</p> <p>c. Could the lab handle quick turnaround samples?</p> <p>d. Overall, is the lab acceptable for AVO analysis?</p>		

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**CHART I-12**

**ORGANIC ANALYSIS BY GC: AVO (8020)**

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ITEM
Additional observations, comments, or problems:

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for phenols sample preparation and analysis?</li> <li>b. Do these SOPs accurately reflect procedures in use?</li> <li>c. Are manufacturers operating manuals readily available to bench chemists?</li> <li>d. Are prenumbered, bound notebooks used for data entry?</li> <li>e. Are all records written in indelible ink?</li> <li>f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</li> <li>g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms?</li> <li>b. Are backup bench chemists available?</li> <li>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</li> </ul>		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is oven temperature stable to $\pm 0.5^{\circ}\text{C}$ or better at desired setting?		
d. Is a 1.8-m x 2-mm ID glass column packed with 1% SP-1240 DA on Supercoport (80/100 mesh) or an equivalent column in use for the determination of underivatized phenols?		
e. Is a flame ionization detector available for the determination of underivatized phenols?		
f. Is nitrogen carrier gas available for use with the FID?		
g. Is a 1.8-m x 2-mm ID glass column packed with 5% OV-17 on Chromosorb W-AW-DMCS (80/100 mesh) or an equivalent column in use for the determination of derivatized phenols?		
h. Is an electron capture detector (ECD) available for the determination of derivatized phenols?		
i. Is 5% methane/95% argon carrier gas available for use with the ECD?		

CHART 1-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>j. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</p> <p>k. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>l. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>m. Has any instrument been modified in any way?</p> <p>n. Is a hood available for sample preparation?</p> <p>o. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>p. Are backup apparatus available?</p>		
<p>Reagents:</p> <p>a. Is reagent water used free from interferents at the MDL of target analytes?</p> <p>c. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?</p> <p>c. Are the following reagents available for use in derivatization:</p> <p>(1) Pentafluorobenzene bromide (<math>\alpha</math>-Bromopentafluorotoluene)?</p>		

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CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
(2) 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane)?  d. Are derivatization reagents prepared fresh weekly and stored at 4°C from light?  e. Are "pesticide quality" or equivalent solvents used for sample analysis?  f. Does the lab have calibration standards for all method specified target analytes?  g. Are calibration standards prepared with 2-propanol as a solvent?  h. Are stock standard solutions stored at 4°C and protected from light?  i. Are stock standard solutions replaced after one year, or sooner if comparison with check standards indicates a problems?  j. Are working standards replaced after six months, or sooner if comparison with calibration standards indicates a problems?  k. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:  a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks? c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte? d. Is one of the calibration standards at a concentration near, but above, the MDL? e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector? f. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range? g. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard? h. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than ±15%?		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
i. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?  j. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation:  a. Are aqueous samples extracted at a pH $\leq 2$ with methylene chloride, using Method 3510A or 3520A?  b. Are solid samples extracted using either Method 3540A or 3550?  c. Are extracts from either Method 3520A or 3550 undergone acid-base partition cleanup, using Method 3650A?  d. Is the extraction solvent exchanged to 2-propanol prior to GC analysis?  e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
Sample Analysis:  a. Is daily calibration performed with a mid-concentration standard prior to sample analysis?  b. Is daily calibration checked at the end of an analysis sequence?		

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ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>c. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds <math>\pm 15\%</math> when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria?</p> <p>d. Are daily retention time windows established for each analyte prior to sample analysis?</p> <p>e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?</p> <p>f. Is solvent flush technique used to inject samples to GC?</p> <p>g. If interferences prevent measurement of peak area during analysis by an FID, is the phenol extract derivatized by pentafluorobenzylbromide (PFB) and the derivatized extract cleaned up using Method 3630A (silica gel cleanup) and analyzed by an ECD?</p> <p>h. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed?</p> <p>i. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?</p> <p>j. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/<math>\mu</math>L in the final extract)?</p>		

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ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
k. If sample extracts are cleaned up with Methods 3630A/3650A, are the associated QC samples processed through the same methods?		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific MDL equal to or lower than the method specified MDL?</p> <p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p>		

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ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the sample analysis halted until the system performance is back in control?</p> <p>g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul>		

CHART I-13

ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>h. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with phenolic surrogates using 2-fluorophenol and 2,4, 6-tribromophenol to encompass the range of temperature used in this method?</p> <p>i. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?</p> <p>j. Are control limits for each surrogate in a given matrix calculated based on the above data?</p> <p>k. Do the control limits fall within the control limits of Method 8270 if applicable?</p> <p>l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p>		

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ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
<p>m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogates for a sample are out of control limits?</p> <p>n. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>o. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p>		

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ORGANIC ANALYSIS BY GC: PHENOLS (8040A)

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ITEM	YES	COMMENT
b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for phenols analysis?</p> <p>b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____</p> <p>c. Could the lab handle quick turnaround samples?</p> <p>d. Overall, is the lab acceptable for phenols analysis?</p>		
Additional observations, comments, or problems:		

**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPS available and adequate for PEST/PCB sample preparation and analysis?</li> <li>b. Do these SOPS accurately reflect procedures in use?</li> <li>c. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>d. Are prenumbered, bound notebooks used for data entry?</li> <li>e. Are all records written in indelible ink?</li> <li>f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</li> <li>g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms?</li> <li>b. Are backup bench chemists available?</li> <li>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</li> </ul>		

**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
d. Does the lab have experienced residue analysis experts on staff?		
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separatory funnels (2,000 mL with Teflon stopcock), Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is gas chromatography equipped with an glass-lined injection port, and an electron capture or electrolytic conductivity detector available?		
d. Is oven temperature stable to $\pm 0.5^{\circ}\text{C}$ or better at desired setting?		
e. Is carrier-gas line equipped with a molecular sieve drying cartridge and a trap for removal of oxygen from the carrier gas?		
f. Is one of the following glass GC column available?		
(1) 1.8-m x 4-mm ID glass, packed with 1.5% SP-2250/1.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent?		
(2) 1.8-m x 4-mm ID glass, packed with 3% OV-1 on Supelcoport (100/120 mesh) or equivalent?		
g. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
<p>h. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>i. Has any instrument been modified in any way?</p> <p>j. Is a hood available for sample preparation?</p> <p>k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>l. Are backup apparatus available?</p> <p>m. Is glassware properly cleaned and finally rinsed with pesticide-quality hexane?</p> <p>n. Is volumetric glassware cleaned with "No Chromix" or equivalent?</p> <p>o. Is heavily contaminated glassware heated in a muffle furnace at 400°C for 15 to 30 minutes?</p> <p>p. Is glassware contaminated with high-boiling-point materials, such as PCBS, heated at 500°C overnight? (Borosilicate glassware shall not be heated above this temperature.)</p> <p>q. Is high temperature treatment on volumetric glassware, glassware with ground joints, or sintered glassware avoid?</p>		
<p>Reagents:</p> <p>a. Is reagent water used free from interferents at the MDL of target analytes?</p>		

**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Are all chemical reagents for pesticide and PCB analyses stored in glass containers?  d. Are "pesticide quality" or equivalent solvents used for pesticide analysis?  e. Are all solvents stored in glass containers and transferred with all glass system?  f. Is 5% methane/95% argon carrier gas available?  g. Are solvent extracted silicon carbide or equivalent used as boiling chips?  h. Does the lab have calibration standards for all method specified target PCBs?  i. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:  a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?  b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
<p>Instrument Calibration and Maintenance:</p> <ul style="list-style-type: none"> <li>a. Is there a calibration protocol readily available to bench chemists?</li> <li>b. Are calibration results kept in permanent logbooks?</li> <li>c. If the GC is not used for a day or more, is the GC column primed or deactivated by injecting a PCB or pesticide standard mixture about 20 times more concentrated than the mid-level standard, prior to instrument calibration?</li> <li>d. Is a calibration blank run following the system prime to ensure no carryover contamination?</li> <li>e. Is a mid-level standard contain only 4,4'-DDT and endrin injected to check the degradation problem at injection port or front of the column prior to calibration?</li> <li>f. If the degradation of either DDT or endrin exceeds 20% (or 15% for capillary column) based on peak areas, is corrective action taken before proceeding with calibration?</li> <li>g. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?</li> <li>h. Is one of the external standards at a concentration near, but above, the MDL?</li> </ul>		

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**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
i. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
j. Is a linear calibration curve with a correlation coefficient $\geq 0.995$ prepared for each analyte?		
k. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?		
l. Is the total area of all peaks measured from the common baseline under all peaks used for quantitation of multiresponse analytes?		
m. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		
n. Is a new calibration curve prepared for any target analyte when the response or the target analyte varies from the predicted response by more than $\pm 5\%$ ?		
o. Is the retention time window established with three injections of all single component standard mixtures and multiple response products throughout the course of a 72-hour period?		
p. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
<p>Sample Preparation:</p> <ul style="list-style-type: none"> <li>a. Are aqueous samples extracted at a neutral, or as is, pH with methylene chloride, using Method 3510 or 3520?</li> <li>b. Are solid samples extracted using either Method 3540 or 3550?</li> <li>c. Is entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples?</li> <li>d. Is sample bottle rinsed with extraction solvent and the rinsate combined with extract?</li> <li>e. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</li> </ul>		
<p>Sample Analysis:</p> <ul style="list-style-type: none"> <li>a. Is daily calibration performed with a mid-concentration standard prior to sample analysis?</li> <li>b. Is daily calibration checked at the end of an analysis sequence?</li> <li>c. If the calibration factor based on daily calibration check at the end of an analysis sequence exceeds <math>\pm 15\%</math> when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples which contain target analytes that exceed the criteria?</li> <li>d. Are daily retention time windows established for each analyte prior to sample analysis?</li> </ul>		

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ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
f. Is the volume of sample injected recorded to the nearest 0.05 $\mu$ L?		
g. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed?		
h. Are peak height measurements used for quantitation when overlapping peaks caused errors in area integration?		
i. If peak detection and identification are prevented due to interference, does the extract routinely undergo a Florisil column cleanup (Method 3620A) and/or sulfur cleanup (Method 3660A) to eliminate interferences?		
j. Is mercury, activated copper powder, or tetrabutylammonium (TBA)-sulfite reagent used for sulfur cleanup?		
k. Is microcoulometric or halogen specific (i.e., electrolytic conductivity) detector used to eliminate interference caused by phthalate esters?		
l. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/ $\mu$ L in the final extract)?		
m. If the early portion of toxaphene chromatogram is interfered with by other substances, is area of the last four peaks in both sample and standard used for quantitation?		

**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
<p>n. If chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, is each peak quantitated separately against appropriate reference materials and reported as individual residues?</p> <p>o. Is the total area of all peaks measured from the common baseline under all peaks used for PCB quantitation?</p> <p>p. Are only those peaks that can be attributed to chlorobiphenyls used for PCB quantitation?</p> <p>q. If there are interference peaks within the Aroclor pattern, is the PCB quantitation determined with three to five major peaks that are <math>\geq 25\%</math> of the height of the largest Aroclor peak in the Aroclor standards?</p> <p>r. Is the amount of Aroclor calculated with the individual response factor for each of the major peaks and are the results of the three to five determinations averaged?</p>		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p>		

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**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
<p>d. Is the lab specific MDL equal to or lower than the method specified MDL?</p> <p>e. Is a separate set of internal QC samples including method blanks, LCS, matrix spikes, matrix spike duplicates and matrix duplicates run for each analytical batch of pesticides or PCB?</p> <p>f. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>g. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p>		

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ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
<p>h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p>		

CHART I-14

ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
<p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>i. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with pesticide surrogates using 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCBP) as specified by the method?</p> <p>j. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?</p> <p>k. Are control limits for each surrogate in a given matrix calculated based on the above data?</p> <p>l. Do the control limits fall within the control limits of Method 8270 if applicable?</p> <p>m. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>n. Are corrective actions of reanalysis or reextraction/reanalysis taken if both surrogates for a sample are out of control limits?</p> <p>o. Are control charts for internal QC data plotted and available to bench chemists?</p>		

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ORGANIC ANALYSIS BY GC: PEST/PCB (8080)

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ITEM	YES	COMMENT
p. Are control limits for internal quality control empirically established and updated on a regular basis?		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		

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**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM	YES	COMMENT
Overall Evaluation:  a. Does the lab have sound technical capability for PEST/PCB analyses?  b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for PEST/PCB analyses?		

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**CHART I-14**

**ORGANIC ANALYSIS BY GC: PEST/PCB (8080)**

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ITEM
Additional observations, comments, or problems:

## CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>General:</p> <p>a. Are written SOPS available and adequate for PAH sample preparation/analysis?</p> <p>b. Do these SOPS accurately reflect procedures in use?</p> <p>c. Are manufacturer's operating manuals readily available to bench chemists?</p> <p>d. Are prenumbered, bound notebooks used for data entry?</p> <p>e. Are all records written in indelible ink?</p> <p>f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</p> <p>g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</p>		
<p>Technical Staff:</p> <p>a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms?</p> <p>b. Are backup bench chemists available?</p> <p>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

Page 2 of 12

ITEM	YES	COMMENT
<p>Apparatus and Facilities:</p> <ul style="list-style-type: none"> <li>a. Is working space adequate and clean?</li> <li>b. Are enough sets of separatory funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?</li> <li>c. Is oven temperature stable to <math>\pm 0.5^{\circ}\text{C}</math> or better at desired setting?</li> <li>d. Is one of the following glass GC column available? <ul style="list-style-type: none"> <li>(1) 1.8-m x 2-mm ID glass column packed with 3% OV-17 on Chromosorb W-AW-DCMS (100/120 mesh) or equivalent?</li> <li>(2) 30-m x 0.25-mm ID SE-54 fused silica capillary column?</li> <li>(3) 30-m x 0.32-mm ID SE-54 fused silica capillary column?</li> </ul> </li> <li>e. If capillary column is in use, is helium used as the carrier gas?</li> <li>f. If packed column is in use, is nitrogen used as the carrier gas?</li> <li>g. Is a flame ionization detector available?</li> <li>h. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</li> </ul>		

## CHART I-15

## ORGANIC ANALYSIS BY GC: PAH (8100)

Page 3 of 12

ITEM	YES	COMMENT
i. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?  j. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?  k. Has any instrument been modified in any way?  l. Is a hood available for sample preparation?  m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?  n. Are backup apparatus available?		
Reagents:  a. Is reagent water used free from interferents at the MDL of target analytes?  b. Are reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Are "pesticide quality" or equivalent solvents used for sample analysis?  d. Does the lab have calibration standards for all method specified target analytes?  e. Are calibration standards prepared with isooctane as a solvent?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>f. Are stock standard solutions stored at 4°C and protected from light?</p> <p>g. Are stock standard solutions replaced after one year, or sooner if comparison with check standards indicates a problem?</p> <p>h. Are working standards replaced after six months, or sooner if comparison with check standards indicates a problem?</p> <p>i. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?</p> <p>b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>d. Is one of the external standards at a concentration near, but above, the MDL?</p> <p>e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?</p> <p>f. Is a linear calibration curve with a correlation coefficient <math>\geq 0.995</math> prepared for each analyte?</p> <p>g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?</p> <p>h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?</p> <p>i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than <math>\pm 15\%</math>?</p> <p>j. Is retention time window established with three injections of all single component standard mixtures and multiple response products throughout the course of a 72-hour period?</p> <p>k. Is retention time window checked on a quarterly basis or whenever a new GC column is installed?</p>		
<p>Sample Preparation:</p> <p>a. Are aqueous samples extracted at a neutral pH with methylene chloride, using Method 3510 or 3520?</p>		

**CHART I-15**

**ORGANIC ANALYSIS BY GC: PAH (8100)**

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ITEM	YES	COMMENT
<p>b. Are solid samples extracted using either Method 3540 or 3550?</p> <p>c. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Is daily calibration performed with a mid-concentration standard prior to sample analysis?</p> <p>b. Is daily calibration checked at the end of an analysis sequence?</p> <p>c. If the calibration factor calculated from daily calibration check at the end of an analysis sequence exceeds ±15% when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes that exceed the criteria?</p> <p>d. Are daily retention time windows established for each analyte prior to sample analysis?</p> <p>e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?</p> <p>f. If peak detection and identification are prevented due to interferences, is the extract undergone Method 3630 (Silica Gel Cleanup)?</p>		

## CHART I-15

## ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
g. If the peak areas exceed the linear range of the system, is the extract diluted and reanalyzed?  h. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration?  i. Are any positive hits confirmed by a second GC column (or by GC/MS if the concentration of each positive hit exceeds 10 ng/ $\mu$ L in the final extract)?		
Quality Control:  a. Are all QC data maintained and available for easy reference and inspection?  b. Is a three-level data review carried out within the lab prior to data release?  c. Is a lab specific MDL empirically established and updated on a semiannually basis?  d. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?  e. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?  (1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>f. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever would be higher?</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>g. Is the performance of extraction, cleanup (when used), analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with one or two surrogates, e.g., 2-fluorobiphenyl &amp; 1-fluoronaphthalene, to encompass the range of temperature used in this method?</p> <p>h. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated to establish control limits, when surrogate data from 25 to 30 samples for each matrix is available?</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
<p>i. Do the control limits fall within those of Method 8270 if applicable?</p> <p>j. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>k. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogates for a sample are out of control limits?</p> <p>l. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>m. Are control limits for internal quality control empirically established and updated on a regular basis?</p> <p>n. Because of coelution problems, is the use of this method avoided and the sample analyzed by either HPLC or GC/MS when the four pairs of compounds listed below are encountered?</p> <p>(1) Anthracene and phenanthrene</p> <p>(2) Chrysene and benzo(a)anthracene</p> <p>(3) Benzo(b) fluoroanthene and benzo(k) fluoranthene</p> <p>(4) Dibenzo(a,h) anthracene and indeno(1,2,3-cd)pyrene</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8200)

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for PAH analysis?</p>		

CHART I-15

ORGANIC ANALYSIS BY GC: PAH (8100)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for PAH analysis?		
Additional observations, comments, or problems:		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

Page 1 of 14

ITEM	YES	COMMENT
General:  a. Are written SOPS available and adequate for HERB sample preparation/analysis?  b. Do these SOPS accurately reflect procedures in use?  c. Are manufacturer's operating manuals readily available to bench chemists?  d. Are prenumbered, bound notebooks used for data entry?  e. Are all records written in indelible ink?  f. Are all errors corrected by drawing a single line through the error with correction written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?  g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff:  a. Do bench chemists appear knowledgeable and experienced in operation of a GC system and interpretation of chromatograms?  b. Are backup bench chemists available?  c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
d. Do bench chemists have proper experience in working with diazomethane which is explosive and carcinogenic?		
<p>Apparatus and Facilities:</p> <p>a. Is working space adequate and clean?</p> <p>b. Is a temperature-programmable gas chromatography equipped with an electron capture detector, microcoulometric detector, or electrolytic conductivity detectors?</p> <p>c. Is oven temperature stable to <math>\pm 0.5^{\circ}\text{C}</math> or better at desired setting?</p> <p>d. Is one of the following glass GC column available?</p> <p>(1) 1.8-m x 4-mm ID glass, packed with 1.5% SP-2250/1.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent?</p> <p>(2) 1.8-m x 4-mm ID glass, packed with 5% OV-210 on Gas Chrom Q (100/120 mesh) or equivalent?</p> <p>(3) 1.98-m x 2-mm ID glass, packed with 0.1% SP-1000 on Carbopack C (80/100 mesh) or equivalent?</p> <p>(4) Is column one used as the primary analytical column and columns two or three as a confirmation column?</p> <p>e. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
f. Is a diazomethane generator available at the lab?		
g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
h. Has any instrument been modified in any way?		
i. Are glassware and glass wool acid rinsed prior to use?		
j. Are boiling chips solvent extracted?		
k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?		
l. Are backup instruments available?		
Reagents:		
a. Is reagent water used free from interferents at the MDL of target analytes?		
b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?		
c. Are pesticide-quality or equivalent solvents (i.e., acetone, methanol, and hexane) used?		
d. Is diethyl ether of pesticide quality or equivalent and free of peroxides used?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>e. Is 20 mL of ethyl alcohol preservative added to each liter of cleaned diethyl ether?</p> <p>f. Is sodium sulfate purified by heating at 400°C for four hours or by precleaning with methylene chloride?</p> <p>g. Is sodium sulfate acidified with sulfuric acid prior to use to avoid reaction with herbicides?</p> <p>h. Are stock standards stored in bottles with Teflon-lined screw caps or crimp tops at 4°C and protected from light?</p> <p>i. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem?</p> <p>j. Are working standards replaced after six months or sooner, if comparison with check standards indicates a problem?</p> <p>k. Does the lab use one or two herbicides, that are not expected to be presented in the sample and that elute over the temperature range of this method, as surrogate(s)?</p> <p>l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are herbicide samples stored at 4°C and extracted within seven days (water) or 14 days (soil) from collection?</p>		

**CHART I-16**

**ORGANIC ANALYSIS BY GC: HERB (8150A)**

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ITEM	YES	COMMENT
b. Are extracts stored under refrigeration and analyzed within 40 days from extraction?		
Instrument Calibration and Maintenance:  a. Is there a calibration protocol readily available to bench chemists?  b. Are calibration results kept in permanent logbooks?  c. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?  d. Is one of the calibration standards at a concentration near, but above, the MDL?  e. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?  f. Is a linear calibration curve with a correlation coefficient $\geq 0.995$ prepared for each analyte?  g. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?  h. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>i. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than <math>\pm 15\%</math>?</p> <p>j. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?</p> <p>k. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?</p>		
<p>Sample Preparation:</p> <p>a. Is the pH of aqueous samples adjusted to <math>&lt;2</math> with sulfuric acid prior to extraction?</p> <p>b. Is diethyl ether of pesticide-quality or equivalent and free of peroxides used for extraction of aqueous samples?</p> <p>c. For soil/sediment samples, is the pH of sample adjusted to two with HCl and monitored and adjusted, if needed, for 15 minutes prior to extraction?</p> <p>d. Are multiple extractions with acetone and diethyl ether used for soil and sediment samples?</p> <p>e. Is cold (<math>4^{\circ}\text{C}</math>) sulfuric acid used to adjust the pH to two prior to solvent cleanup?</p> <p>f. Is acidified sodium sulfate used to dry the diethyl ether for a minimum of two hours prior to esterification?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>g. Is a bubble method or a Diazald kit method used at the lab to generate diazomethane?</p> <p>h. Are the following precautions taken during esterification with diazomethane?</p> <p>(1) Use a safety screen?</p> <p>(2) Use mechanical pipetting aides?</p> <p>(3) Do not heat above 90°C?</p> <p>(4) Avoid grinding surfaces, ground glass joint, sleeve bearing, glass stirrers?</p> <p>(5) Store away from alkali metals?</p> <p>(6) Avoid contact with copper powder, calcium chloride, and boiling chips?</p> <p>i. Is methylated extracts analyzed immediately to minimize trans-esterification and other potential reactions?</p>		
<p>Sample Analysis:</p> <p>a. Is GC column 1 selected for majority of herbicide analysis, except for Dalapon which is analyzed with GC column 3?</p> <p>b. Is daily calibration performed with a mid-concentration standard at the beginning and the end of an analysis sequence?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
c. If the calibration factor calculated from injection of a mid-concentration standard at the end of an analysis sequence exceeds $\pm 15\%$ when compared with the initial standard of the analysis sequence, is the GC system recalibrated and reanalysis performed for all samples, in the sequence, which contain target analytes exceed the criteria?		
d. Are daily retention time windows established for each analyte prior to sample analysis?		
e. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?		
f. Have calibration standards undergone the same hydrolysis and esterification processes as the samples?		
g. If calibration is done with standards made from methyl ester compounds, is the final concentration corrected for molecular weight of methyl ester versus the acid herbicides?		
h. If a peak response exceeds the linear range of the system, is a dilution performed on a second aliquot of the sample which has been properly sealed and stored prior to use?		
i. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration?		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
j. Is further extract cleanup routinely conducted if interferences prevent peak detection and identification?		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific MDL equal to or lower than the method specified MDL?</p> <p>e. Are GC/MS techniques routinely used to confirm positive hits?</p> <p>f. If GC/MS fails, are additional steps including alternative packed or capillary GC columns or additional cleanup routinely taken for qualitative confirmation?</p> <p>g. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>h. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent of calibration standards analyzed for each batch?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>i. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>j. Is the performance of extraction, cleanup, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?</p> <p>k. Are the average percent recovery and standard deviation of the percent recovery for each surrogate calculated, when surrogate data from 25 to 30 samples for each matrix is available?</p> <p>l. Are control limits for each surrogate in a given matrix calculated based on the above data?</p>		

CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
<p>m. Do the control limits fall within the control limits of Method 8270 if applicable?</p> <p>n. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>o. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?</p> <p>p. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>q. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		

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CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM	YES	COMMENT
Waste Disposal:  a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?  b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation:  a. Does the lab have sound technical capability for HERB analysis?  b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for HERB analysis?		

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CHART I-16

ORGANIC ANALYSIS BY GC: HERB (8150A)

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ITEM
Additional observations, comments, or problems:

CHART I-17

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS:

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ITEM	YES	COMMENT
Is the MS capable of scanning from 35 to 450 amu every seven seconds or less?		
Is tuning compound, FC-43, used to verify mass calibration?		
Do the mass spectra of BFP and DFTPP meet all criteria before each batch of volatile and semivolatile samples is run?		
Are standards containing all of the analytes of interest analyzed to verify response factors and update retention time?		
Is glassware for organics solvent rinsed or heated to a minimum of 300°C to vaporize any organics in a muffle furnace after careful cleaning?		
Is this high temperature treatment avoided for volumetric glassware, glassware with ground joints, or sintered glassware?		
Is glassware sealed and stored in a clean environment?		
Are magnetic tapes stored in a secure area?		
Are extensive in-house replacement parts available?		
Are manufacturer's operating manuals readily available to bench chemists?		
Is there a calibration protocol available to the bench chemists?		
Are calibration results kept in a permanent logbook?		

CHART I-17

GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS:

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ITEM	YES	COMMENT
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Has the instrument been modified in any way?		
Are the instruments properly vented?		
Is a 5-point calibration used?		
Are continuing calibration checks done on a 12-hour basis?		
Are system performance response factors checked on a 12-hour basis?		
Are BFB and DFTPP tuning checks done on a 12-hour basis?		
Is low-level method routinely used for environmental soil/sediment samples?		
For tentatively identified compounds, are library searches done for the ten volatile organics and the 20 semivolatile organics of highest concentration?		
Are surrogate recoveries run on each sample?		
Is a corrective action taken if surrogate recoveries exceed QC limits?		
Is a method blank included with each batch of samples and carried through the entire preparation and analysis?		
Is a lab duplicate run at a rate of 5% or one per batch, whichever is greater?		

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GENERAL QA/QC FOR ORGANIC ANALYSIS BY GC/MS:

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ITEM	YES	COMMENT
Is a corrective action taken if matrix spike recoveries exceed QC limits?		
Is a spiked sample run at a rate of 5% or one per batch, whichever is greater?		
Is an LCS analyzed with every tenth sample or each batch?		
Additional observations, comments, or problems:		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>General:</p> <p>a. Are written SOPS available and adequate for VOA sample preparation/analysis?</p> <p>b. Do these SOPS accurately reflect procedures in use?</p> <p>c. Are all target analytes, at a minimum, listed in Table 2 of Method 8240A routinely analyzed at the lab?</p> <p>d. Are manufacturer's operating manuals readily available to bench chemists?</p> <p>e. Are prenumbered, bound notebooks used for data entry?</p> <p>f. Are all records written in indelible ink?</p> <p>g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</p> <p>h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</p>		
<p>Technical Staff:</p> <p>a. Do bench chemists appear knowledgeable and experienced in operation of a purge-and-trap and GC/MS system and in interpretation of chromatograms and mass spectra?</p> <p>b. Are backup bench chemists available?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities:  a. Is working space adequate and clean?  b. Does the lab have adequate air handling system to avoid cross contamination of samples?  c. Is a temperature-programmable gas chromatography equipped with a purge-and-trap device available?  d. Is oven temperature stable to $\pm 0.5^{\circ}\text{C}$ or better at desired setting?  e. Is a GC column of 6-ft x 0.1-in ID glass, packed with 1% SP-1000 on Carbowack-B (60/80 mesh) or equivalent, available?  f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?  g. Are enough sets of purge-and-trap devices available for all samples in an analytical batch?  h. Is the mass spectrometer capable of scanning from 35 - 260 amu every three seconds or less, using 70-volt electron energy in the electron impact mode?  i. Is a computer data system that allows continuous acquisition and storage on machine-readable media of all mass spectra available?		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>j. Is the most recent version of the EPA/NIST Mass Spectral Library available?</p> <p>k. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>l. Is sample preparation conducted in a hood?</p> <p>m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>n. Are backup instruments available?</p>		
<p>Reagents:</p> <p>a. Is reagent water used free from interferents at the MDL of target analytes?</p> <p>b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?</p> <p>c. For standard preparation, is a waiting period of ten minutes allowed for drying the alcohol-wetted surface before measuring the weight of methanol to the nearest 0.1 mg?</p> <p>d. Are stock standards stored in bottles with minimal headspace and Teflon-lined screw cap at -10 to -20°C and protected from light?</p> <p>e. Are stock standards replaced after six months, or sooner if comparison with check standards indicates a program?</p>		

CHART I-18

ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
f. Are stock standards for target analytes of low boiling points (<30°C) and high reactivity prepared fresh every two months or sooner?		
g. Are secondary standards stored with minimal headspace and check frequently for degradation or evaporation?		
h. Is 2 mL of GC/MS system tuning standard, containing 25 ng/μL of 4-bromofluorobenzene (BFB) in methanol injected or purged for hardware tuning?		
i. Are method recommended surrogates, toluene-d <sub>8</sub> , 4-bromofluorobenzene, and 1,2-dichloroethane-d <sub>4</sub> spiked into each sample undergoing GC/MS analysis?		
j. Are method recommended internal standards, bromochloromethane, 1,4-dichlorobenzene, and chlorobenzene-d <sub>5</sub> or other compounds with retention times similar to the compounds being detected by GC/MS?		
k. For the initial calibration, are aqueous calibration standards, at a minimum of five concentrations, prepared fresh and discarded after one hour, unless properly sealed in a vial and stored at 4°C with no headspace (up to one week)?		
l. Are method recommended matrix spike standards (1,1-dichloroethene, tri-chloroethene, chlorobenzene, toluene, and benzene in methanol at 25 μg/mL) available?		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>m. Are all non-aqueous standard solutions stored at -10 to -20°C in screw-cap amber bottles with Teflon liners?</p> <p>n. Are volatile organic standards stored in a separated freezer/refrigerator from samples or other standards?</p> <p>o. Is "purge-and-trap", "pesticide quality", or equivalent methanol stored away from other solvents?</p> <p>p. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are volatile organic samples stored at 4°C in separate refrigerators from other samples?</p> <p>b. Are low concentration volatile organic samples stored separately from high concentration volatile organic samples?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Is the trap of a purge-and-trap device conditioned overnight at 180°C in the purge mode with an inert gas flow of at least 20 mL/min?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT																				
d. Prior to use, is the trap conditioned daily for 10 minutes while backflushing at 180°C with the column at 220°C?																						
e. Is manufacturer's recommendations used for conditioning of the purge-and trap device?																						
f. Initial Calibration:																						
(1) Is each GC/MS system hardware-tuned to meet the criteria for 50-ng injection or purging of BFB prior to sample analysis?																						
<table><tr><th>Mass</th><th>Ion Abundance Criteria</th></tr><tr><td>50</td><td>15% to 45% of mass 95</td></tr><tr><td>75</td><td>30% to 60% of mass 95</td></tr><tr><td>95</td><td>base peak, 100% relative abundance</td></tr><tr><td>96</td><td>5% to 9% of mass 95</td></tr><tr><td>173</td><td>0% to &lt;2% of mass 174</td></tr><tr><td>174</td><td>&gt;50% of mass 95</td></tr><tr><td>175</td><td>5% to 9% of mass 174</td></tr><tr><td>176</td><td>&gt;95% but &lt;101% of mass 174</td></tr><tr><td>177</td><td>5% to 9% of mass 176</td></tr></table>	Mass	Ion Abundance Criteria	50	15% to 45% of mass 95	75	30% to 60% of mass 95	95	base peak, 100% relative abundance	96	5% to 9% of mass 95	173	0% to <2% of mass 174	174	>50% of mass 95	175	5% to 9% of mass 174	176	>95% but <101% of mass 174	177	5% to 9% of mass 176		
Mass	Ion Abundance Criteria																					
50	15% to 45% of mass 95																					
75	30% to 60% of mass 95																					
95	base peak, 100% relative abundance																					
96	5% to 9% of mass 95																					
173	0% to <2% of mass 174																					
174	>50% of mass 95																					
175	5% to 9% of mass 174																					
176	>95% but <101% of mass 174																					
177	5% to 9% of mass 176																					
(2) Is the initial calibration performed with a minimum of five concentration levels for each target analyte?																						
(3) Is one of the calibration standards at a concentration near, but above, the MDL?																						
(4) Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?																						

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>(5) Is a system performance check made with five System Performance Check Compounds (SPCCs) for a minimal average response factor (RF) of 0.300 for each SPCC (0.250 for bromoform)?</p> <p>The SPCCs are:  Chloromethane,  1,1-Dichloroethane,  Bromoform,  1,1,2, 2-Tetrachloroethane, and  Chlorobenzene.</p> <p>(a) Chloromethane will be lost if the purge flow is too fast.</p> <p>(b) Bromoform will be purged very poorly if purge flow is too slow. Cold spots and/or active sites may adversely affect response.</p> <p>(c) Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines and/or active sites.</p> <p>(6) Is percent relative standard deviation for each Calibration Check Compound (CCC), less than 30%, based on the RFs from the initial calibration?</p> <p>The CCCs are:  1,1-Dichloroethene,  Chloroform,  1,2-Dichloropropane,  Toluene,  Ethylbenzene, and  Vinyl chloride.</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>g. Daily Calibration:</p> <p>(1) Is each GC/MS system hardware-tuned to meet BFB tuning criteria for each 12-hour shift prior to sample analysis?</p> <p>(2) Is the initial calibration curve for each target analyte checked and verified by checking SPCC and CCC of a midpoint calibration standard every 12-hour shift?</p> <p>(3) Do the RFs of SPCCs meet the initial SPCC criteria for each 12-hour shift?</p> <p>(4) Is the percent difference on RFs less than 25% for any one CCC?</p> <p>(5) If the criteria in (3) and (4) are not met, is corrective action taken to solve possible problems such as standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or GC system?</p> <p>(6) If no source of problem can be determined after corrective action has been taken, is a new 5-point calibration generated?</p> <p>(7) Are the retention times of the internal standards in the check calibration standard within 30 seconds from the last daily calibration check (12 hour)?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>(8) Is the response of the internal standards in the check calibration standard within a factor of two (-50% to +100%) from the last daily calibration standard check (12 hour)?</p> <p>(9) If the criteria in (7) and (8) are not met, is the mass spectrometer inspected and corrected?</p> <p>(10) If corrections are made, is reanalysis conducted for samples analyzed while the system was malfunctioning?</p>		
<p>Sample Preparation:</p> <p>a. Are purge-and-trap (Method 5030) used for the extraction and injection of standards and samples?</p> <p>b. Before initial use, is the trap conditioned overnight at 180°C by back flushing with an inert gas flow of at least 20 mL per minute?</p> <p>c. Prior to daily use, is the trap conditioned for 10 minutes at 180°C with back flushing?</p>		
<p>Sample Analysis:</p> <p>a. Are all samples and standard solutions allowed to warm to ambient temperature before analysis?</p> <p>b. Is the flow rate of helium purge for best response for chloromethane and bromoform? (<math>\approx</math>30-40 mL per minute)</p>		

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**ORGANIC ANALYSIS BY GC/MS: VOA (8240A)**

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ITEM	YES	COMMENT
c. If a second analysis is needed from sample stored in a syringe, is the analysis completed within 24 hours?		
d. Is the purging chamber washed with two 5-mL flushes of reagent water or methanol followed by reagent water to avoid carryover?		
e. If the concentration of analytes in a sample exceeds the calibration ranges, is the sample diluted and reanalyzed? (Diluted to upper half of curve.)		
f. If sample dilution is needed, is an aliquot of sample which is not less than 1 mL used for dilution and the mixture only inverted and shake three times to minimize loss?		
g. Is proper dilution conducted to keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of calibration curve?		
h. Is secondary ion quantitation used only when there are sample interferences with primary ion quantitation?		
i. Is there a method blank analyzed after a sample that has saturated ions from a compound?		
j. If the blank is not free of interferences, is the system cleaned prior to resuming sample analysis?		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>k. Are sediment/soil and waste samples screened by headspace (Method 3810) or hexadecane extraction (Method 3820) to determine whether the high-level method should be used?</p> <p>l. Is the low-level method used for samples containing individual compounds of &lt;1 mg/kg and the high-level method used only for samples with an expected concentration of &gt;1 mg/kg?</p> <p>m. Is a 5-g sample used if the expected concentration is &lt;0.1 mg/kg or a 1-g sample for expected concentration between 0.1 and 1 mg/kg?</p> <p>n. Is a heated purge calibration curve (40°C) prepared and used for the quantitation of all low-level sediment/soil samples?</p> <p>o. Do the standards and method blank for high-level method contain 100 µL of methanol to simulate the sample conditions?</p>		
<p>Data Interpretations:</p> <p>a. Is the relative retention window (RRT) for each compound set at ±0.06 RRT units of the RRT of the standard compound analyzed within the same 12 hours as the sample?</p> <p>b. Are major ions in the standard mass spectra at a relative intensity &gt;10% present in the sample spectra?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>c. Do the relative intensities of the major ions agree within 20% between the standard and sample spectra?</p> <p>d. Are molecular ions present in the reference spectrum also present in the sample spectrum?</p> <p>e. Is the lab capable to conduct a computer library search to identify and quantify tentatively identified compounds (TICs)?</p> <p>f. Is the identification of TICS determined only after visual comparison of a sample with the closest library search?</p> <p>g. Is the internal standard of nearest retention time of that of a given compound used for quantification?</p>		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Are lab specific MDL and PQL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific PQL equal to or lower than the method specified PQL?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (If a lab analyzes one to ten samples per month, at least one spiked sample per month is required.)</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?		
(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?		
(3) If it is not possible to determine the background concentration, is the spike concentration <ul style="list-style-type: none"><li>- the regulatory limit, if any; or</li><li>- the larger of either five times the expected background or LCS concentrations?</li></ul>		
(4) For other matrices, is the spike concentration at ten times the estimated quantitation limit?		
(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
<p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>h. Is the performance of purge-and-trap, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?</p> <p>i. Are control limits for internal quality control empirically established and updated on a regular basis?</p> <p>j. Are lab's control limits for surrogates within the method specified limits?</p> <p>k. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>l. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated once a minimum of 30 samples of same matrix have been analyzed?</p> <p>m. Is the method accuracy for each matrix studied assessed and recorded after the analysis of five spiked samples?</p> <p>n. Is the accuracy assessment for each analyte updated after each five to ten new accuracy measurements?</p> <p>o. Are control charts for internal QC data plotted and available to bench chemists?</p>		

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ORGANIC ANALYSIS BY GC/MS: VOA (8240A)

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ITEM	YES	COMMENT
p. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		

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**ORGANIC ANALYSIS BY GC/MS: VOA (8240A)**

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ITEM	YES	COMMENT
Overall Evaluation:  a. Does the lab have sound technical capability for VOA analysis?  b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for VOA analysis?		

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**ORGANIC ANALYSIS BY GC/MS: VOA (8240A)**

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ITEM
Additional observation, comments, or problems:

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>General:</p> <p>a. Are written SOPs available and adequate for BNA sample preparation/analysis?</p> <p>b. Do these SOPs accurately reflect procedures in use?</p> <p>c. Are all target analytes, at a minimum, listed in Table 2 of Method 8270A routinely analyzed at the lab?</p> <p>d. Are manufacturer's operating manuals readily available to bench chemists?</p> <p>e. Are prenumbered, bound notebooks used for data entry?</p> <p>f. Are all records written in indelible ink?</p> <p>g. Are all errors corrected by drawing a single line through the error with correction written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</p> <p>h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</p>		
<p>Technical Staff:</p> <p>a. Do bench chemists appear knowledgeable and experienced in operation of a GC/MS system and in interpretation of chromatograms and mass spectra?</p> <p>b. Are backup bench chemists available?</p>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		
Apparatus and Facilities:  a. Is working space adequate and clean?  b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?  c. Is a temperature-programmable gas chromatography equipped available?  d. Is oven temperature stable to $\pm 0.5^{\circ}\text{C}$ or better at desired setting?  e. Is the following GC column available?  30-m x 0.25-mm ID (or 0.32-mm ID) 1- $\mu\text{m}$ film thickness silicone-coated fused silica capillary column or equivalent.  f. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?  g. Is the mass spectrometer capable of scanning from 35 - 500 amu every one second or less, using 70-volt electron energy in the electron impact mode?  h. Is a computer data system that allows continuous acquisition and storage on machine-readable media of all mass spectra available?		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
i. Is the most recent version of the EPA/ NIST Mass Spectral Library available?  j. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?  k. Has any instrument been modified in any way?  l. Is sample preparation conducted in a hood?  m. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?  n. Are backup instruments available?		
Reagents:  a. Is reagent water used free from interferents at the MDL of target analytes?  b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Are stock standards stored in bottles with minimal headspace and Teflon line screw-cap at 4°C and protected from light?  d. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a program?		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
e. Is a GC/MS system tuning standard, containing 50 ng/μL of decafluorotriphenylphosphine (DFTPP) in methylene chloride, prepared?		
f. Are method recommended surrogates, phenol-d <sub>5</sub> , 2-fluorophenol, 2,4,6-tri-bromophenol, nitrobenzene-d <sub>5</sub> , 2-fluoro-biphenyl, and d-terphenyl <sub>14</sub> -d <sub>14</sub> into each sample undergoing GC/MS analysis?		
g. Are method recommended internal standards, 1,4-dichlorobenzene-d <sub>4</sub> , naphthalene-d <sub>8</sub> , acenaphthene-d <sub>10</sub> , phenanthrene-d <sub>10</sub> , chrysene-d <sub>12</sub> , and perylene-d <sub>12</sub> or other compounds with retention times similar to the compounds (within ±20% of internal standards') being detected by GC/MS?		
h. Are daily calibration standards, at a minimum of five concentrations, stored at 4°C and freshly prepared weekly or sooner if comparison with check standards indicates a problem?		
i. Are method recommended matrix spike standards (pentachlorophenol, phenol, 2-chlorophenol, 4-nitrophenol, 4-chloro-3-methylphenol, 1,2,4-trichlorobenzene, acenaphthene, pyrene, 2,4-dinitrotoluene, N-nitroso-di-n-propylamine, and 1,4-dichlorobenzene) in methanol available?		
j. Are all non-aqueous standard solutions stored at -10°C to -20°C in screw-cap amber bottles with Teflon liners?		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
k. Are "pesticide quality" or equivalent methanol stored away from other solvents?  l. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:  a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?  b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?  c. Are all samples and sample extracts stored in the dark at 4°C?		
Instrument Calibration and Maintenance:  a. Is there a calibration protocol readily available to bench chemists?  b. Are calibration results kept in permanent logbooks?  c. Initial Calibration:  (1) Is each GC/MS system hardware-tuned to meet the criteria for 50-ng injection or purging of DFTPP prior to sample analysis?		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM		YES	COMMENT
<u>Mass</u>	<u>Ion Abundance Criteria</u>		
51	30% to 60% of mass 198		
68	<2% of mass 69		
70	<2% of mass 69		
127	40% to 60% of mass 198		
197	<1% of mass 198		
198	Base peak, 100% relative abundance		
195	5% to 9% of mass 198		
275	10% to 30% of mass 198		
365	>1% of mass 198		
441	Present but less than mass 443		
442	>40% of mass 198		
443	17% to 23% of mass 442		
(2) Does the DFTPP tuning standard also contain 50 ng/μL each of 4,4'-DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance? (<20% of DDT degradation and no visible peak tailing for benzidine and pentachlorophenol.)			
(3) Is the initial calibration performed with a minimum of five concentration levels for each target analyte?			
(4) Is one of the calibration standards at a concentration near, but above, the MDL?			
(5) Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?			

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>(6) Is a system performance check made with four System Performance Check Compounds (SPPCs) for a minimal average response factor (RF) of 0.050 for each compound?</p> <p>The SPCCS are:  N-nitroso-di-n-propylamine,  hexachlorocyclopentadiene,  2,4-dinitrophenol, and  4-nitrophenol.</p> <p>(a) Degradation of DDT to DDE and DDD should not exceed 20%.</p> <p>(b) Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.</p> <p>(7) Is percent relative standard deviation for each Calibration Check Compound (CCC), less than 30%, based on the RFs from the initial calibration?</p> <p>The CCCs are:  4-chloro-3-methylphenol,  2,4-dichlorophenol,  2-nitrophenol,  phenol,  pentachlorophenol,  2,4, 6-trichlorophenol,</p> <p>acenaphthene,  1,4-dichlorobenzene,  hexachlorobutadiene,  N-nitroso-di-n-phenylamine,  di-n-octylphthalate,  fluoranthene,  benzo(a)pyrene.</p>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>d. Daily Calibration:</p> <p>(1) Is each GC/MS system hardware-tuned to meet DFTPP tuning criteria for each 12-hour shift prior to sample analysis?</p> <p>(2) Is the initial calibration curve for each target analyte checked and verified by checking SPCC and CCC of a midpoint calibration standard every 12-hour shift?</p> <p>(3) Do the RFs of SPCCs meet the initial SPCC criteria for each 12-hour shift?</p> <p>(4) Is the percent difference on RFs less than 30% for any one CCC?</p> <p>(5) If the criteria in (3) and (4) are not met, is corrective action taken to solve possible problems such as standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or GC system?</p> <p>(6) If no source of problem can be determined after corrective action has been taken, is a new five-point calibration generated?</p> <p>(7) Are the retention times of the internal standards in the check calibration standard within 30 seconds from the last daily calibration check (12 hours)?</p>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>(8) Is the response of the internal standards in the check calibration standard within a factor of two (-50% to +100%) from the last daily calibration standard check (12 hours)?</p> <p>(9) If the criteria in (7) and (8) are not met, is the mass spectrometer inspected and corrected?</p> <p>(10) If corrections are made, is reanalysis conducted for samples analyzed while the system was malfunctioning?</p> <p>e. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?</p> <p>f. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?</p>		
<p>Sample Preparation:</p> <p>a. Are samples extracted by Methods 3510, 3520, 3540, 3550, or 3580 prior to analysis?</p> <p>b. Are proper extract cleanup methods routinely used prior to analysis?</p> <p>c. Is direct injection used only for samples with concentrations in excess of 10,000 µg/L?</p>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>Sample Analysis:</p> <ul style="list-style-type: none"> <li>a. Is the extract screened on a GC/FID or GC/PID using the same type of capillary column to minimize contamination of GC/MS system from unexpected high concentrations of organic compounds?</li> <li>b. If the concentration of analytes in a sample exceeds the calibration ranges, is the sample diluted and reanalyzed?</li> <li>c. Is additional internal standard added to the diluted extract to maintain the required 40 ng/<math>\mu</math>L of each internal standard in the extract volume?</li> <li>d. Is secondary ion quantitation used only when there are sample interferences with primary ion quantitation?</li> <li>e. Is there a method blank analyzed after a sample that has saturated ions from a compound?</li> <li>f. If the blank is not free of interferences, is the system cleaned prior to resuming sample analysis?</li> </ul>		
<p>Data Interpretations:</p> <ul style="list-style-type: none"> <li>a. Is the relative retention window (RRT) for each compound set at <math>\pm 0.06</math> RRT units of the RRT of the standard compound analyzed within the same 12 hours as the sample?</li> <li>b. Are major ions in the standard mass spectra at a relative intensity <math>&gt;10\%</math> present in the sample spectra?</li> </ul>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
c. Do the relative intensities of the major ions agree within 20% between the standard and sample spectra?  d. Are molecular ions present in the reference spectrum also present in the sample spectrum?  e. Is the lab capable to conduct a computer library search to identify and quantify tentatively identified compounds (TICs)?  f. Is the identification of TICs determined only after visual comparison of a sample with the closest library search?  g. Is the internal standard of nearest retention time of that of a given compound used for quantification?		
Quality Control:  a. Are all QC data maintained and available for easy reference and inspection?  b. Is a three-level data review carried out within the lab prior to data release?  c. Are lab specific MDL and PQL empirically established and updated on a semiannually basis?  d. Is the lab specific PQL equal to or lower than the method specified PQL?  e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the sample analysis halted until the system performance is back in control?</p> <p>g. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples? (If a lab analyzes one to ten samples per month, at least one spiked sample per month is required.)</p> <p>(1) If the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limit or one to five times higher than the background concentration, whichever concentration would be higher?</p>		

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ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 10 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p> <p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>h. Is the performance of sample extraction, analytical system, and the effectiveness of the method in dealing with sample matrix monitored by spiking each sample, standard, and blank with surrogates which encompass the method specified temperature range?</p>		

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CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
i. Are control limits for internal quality control empirically established and updated on a regular basis?		
j. Are lab's control limits for surrogates within the method specified limits?		
k. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
l. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated once a minimum of 30 samples of same matrix have been analyzed?		
m. Is the method accuracy for each matrix studied assessed and recorded after the analysis of five spiked samples?		
n. Is the accuracy assessment for each analyte updated after each five to ten new accuracy measurements?		
o. Are control charts for internal QC data plotted and available to bench chemists?		
p. Are corrective actions of reanalysis or reextraction/reanalysis taken if any surrogates for a sample are out of control limits?		

CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for BNA analysis?</p>		

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CHART I-19

ORGANIC ANALYSIS BY GC/MS: BNA (8270A)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for BNA analysis?		
Additional observations, comments, or problems:		

CHART I-20

ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)

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ITEM	YES	COMMENT
Is a HRGC/LRMS system available for Method 8280?		
Does the lab have a HRGC/HRMS system?		
Is the column oven temperature programmable?		
Is the GC column 60-m long x 0.025-cm ID glass or fused silica, coated with a 0.2 micron film of SP-2330?		
Is the MS low or high resolution with an ion source of 70 volts (nominal)?		
Is a data system interfaced with the mass spectrometer?		
Is the mass spectrometer capable of selected ion monitoring (SIM)?		
If operating conditions such as GC column have changed, has the acceptance criteria for the start up QC been met?		
Are all samples preserved by cooling at 4°C?		
Are all samples extracted within seven days of collection and analyzed within 40 days?		
Is the standard 2,3,7,8-TCDD available?		
Is labeled 2,3,7,8-TCDD available (either $^{13}\text{C}_{14}$ or $^{13}\text{C}_{12}$ ) ?		
Is a record of standard preparation available?		
Are stock standard solutions stored in Teflon sealed screw cap bottles, at 4°C, protected from light?		

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**CHART I-20**

**ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)**

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ITEM	YES	COMMENT
Are stock standard solutions prepared fresh every six months?		
Is a standard curve available?		
Is a method blank included with each sample batch and carried through the entire preparation and analysis?		
Is a lab duplicate run at a rate of 5% or one per batch, whichever is greater?		
Is a spiked sample run at a rate of 5% or one per batch, whichever is greater?		
Is an LCS analyzed with every tenth sample?		
Are results for LCSs charted?		
Are control limits for LCSs established?		
Are charts for LCSs current?		
Are results for spiked sample charted?		
Are control limits established for spiked samples?		
Are charts for spiked samples current?		
Is a temperature controlled ( $\pm 2^{\circ}\text{C}$ ) hot water bath available?		

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**CHART I-20**

**ORGANIC ANALYSIS BY GC/MS: DIOXINS (8280)**

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ITEM
Additional observations, comments, or problems:

**CHART I-21**

**ORGANIC ANALYSIS BY HPLC: PAH (8310)**

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ITEM	YES	COMMENT
General:  a. Are written SOPs available and adequate for PAH sample preparation/analysis?  b. Do these SOPs accurately reflect procedures in use?  c. Are manufacturer's operating manuals readily available to bench chemists?  d. Are prenumbered, bound notebooks used for data entry?  e. Are all records written in indelible ink?  f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?  g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff:  a. Do bench chemists appear knowledgeable and experienced in operation of an HPLC and interpretation of chromatograms?  b. Are backup bench chemists available?  c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
Apparatus and Facilities:		
a. Is working space adequate and clean?		
b. Are enough sets of separator funnels, continuous liquid-liquid extractors, Soxhlet extractors, and Kuderna-Danish apparatuses available for simultaneous extraction of all batch samples?		
c. Is an HPLC equipped with a pump capable of achieving 4,000 psi available?		
d. Can the pump produce a gradient?		
e. Is a fluorescence detector for excitation at 280 nm and emission greater than 389 nm cutoff available?		
f. Is a UV detector at 254 nm coupled to the fluorescence detector available?		
g. Is a reverse phase column, HC-ODS Si-X, 5-micron particle size diameter, in a 250-mm x 2.6-mm ID SS column or equivalent available?		
h. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?		
i. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
j. Has any instrument been modified in any way?		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
k. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?  l. Are backup apparatus available?		
Reagents:  a. Is reagent water used free from interferents at the MDL of target analytes?  b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Is "HPLC grade" or equivalent solvent, acetonitrile, used for PAH analysis?  d. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
Sample Handling and Storage:  a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?  b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?  c. Are all samples and sample extracts stored in the dark at 4°C?		

CHART I-21

ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Are stock standards stored in bottles with Teflon-lined screw caps or crimp tops at 4°C and protected from light?</p> <p>d. Are stock solutions replaced after one year, or sooner if comparison with check standards indicates a problem?</p> <p>e. Are working standards replaced after six months or sooner, if comparison with check standards indicates a problem?</p> <p>f. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?</p> <p>g. Is one of the calibration standards at a concentration near, but above, the MDL?</p> <p>h. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?</p> <p>i. Is a linear calibration curve with a correlation coefficient <math>\geq 0.995</math> prepared for each analyte?</p>		

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**ORGANIC ANALYSIS BY HPLC: PAH (8310)**

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ITEM	YES	COMMENT
j. Is an average calibration factor used only when the percent relative standard deviation of the calibration factor is less than 20% over the working range?		
k. Is the calibration curve or factor verified at the beginning and end of each analysis sequence with a mid-concentration standard?		
l. Is a new calibration curve prepared for any target analyte when the response for the target analyte varies from the predicted response by more than 15%?		
m. Is the retention time window established with three injections of all target analytes throughout the course of a 72-hour period?		
n. Is the retention time window checked on a quarterly basis or whenever a new GC column is installed?		
Sample Preparation:		
a. Are aqueous samples extracted at a neutral, or as is, pH with methylene chloride, using Method 3510 or 3520?		
b. Are solid samples extracted using either Method 3540 or 3550?		
c. Is the entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples?		
d. Is the sample bottle rinsed with extraction solvent and the rinsate combined with extract?		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
<p>e. Is the extraction solvent exchanged to acetonitrile and concentrated to 1 mL with Kuderna-Danish apparatuses and micro-Snyder column prior to HPLC analysis?</p> <p>f. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Is the HPLC elution isocratic with acetonitrile/water (4:6 by volume) for five minutes, then linear gradient to 100% acetonitrile for 25 minutes?</p> <p>b. Is a daily calibration performed with a mid-concentration standard prior to analysis?</p> <p>c. Are daily retention windows established for each analyte prior to sample analysis?</p> <p>d. If the peak areas/heights exceed the linear range of the system, is the extract diluted and reanalyzed?</p> <p>e. Is peak height measurement used for quantitation when overlapping peaks caused errors in area integration?</p>		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p>		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
<p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific MDL equal to or lower than the method specified MDL?</p> <p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>g. Does the matrix spike solution contain all target analytes?</p>		

**CHART I-21**

**ORGANIC ANALYSIS BY HPLC: PAH (8310)**

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ITEM	YES	COMMENT
<p>h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p> <p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p>		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?		
i. Does the lab use one or two analytes which are not expected to be presented in the sample as surrogates? (e.g., decafluorobiphenyl or other PAHs which encompass the retention time ranges.)		
j. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated when surrogate data from 25 to 30 samples for each matrix is available?		
k. Are control limits for each surrogate in a given matrix calculated based on the above data?		
l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?		
m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?		
n. Are control charts for internal QC data plotted and available to operators?		
o. Are control limits for internal quality control empirically established and updated on a regular basis?		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for PAH analysis?</p>		

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ORGANIC ANALYSIS BY HPLC: PAH (8310)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for PAH analysis?		
Additional observations, comments, or problems:		

**CHART I-22**

**ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)**

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for explosives sample preparation and analysis?</li> <li>b. Are the SOPs consistent with the EPA's draft SW-846 Method 8330, Revision 0, November 1992?</li> <li>c. Do these SOPs accurately reflect procedures in use?</li> <li>d. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>e. Are prenumbered, bound notebooks used for data entry?</li> <li>f. Are all records written in indelible ink?</li> <li>g. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error so that it remains legible, and initialed and dated by the responsible individual?</li> <li>h. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in operation of an HPLC and interpretation of chromatograms?</li> <li>b. Are backup bench chemists available?</li> </ul>		

**CHART I-22**

**ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)**

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ITEM	YES	COMMENT
c. Are bench chemists? performance audited and approved prior to work without close supervision by a senior chemist?		
<p>Apparatus and Facilities:</p> <p>a. Is working space adequate and clean?</p> <p>b. Is an HPLC equipped with a pump capable of achieving 4,000 psi, a 100 <math>\mu</math>L loop injector, and 254-nm UV detector available?</p> <p>c. Is the detector capable to achieve a stable baseline at 0.001 absorbance units full scale?</p> <p>d. Are the following HPLC columns available?</p> <p style="padding-left: 40px;">(1) C-18 reverse phase HPLC column, 25-cm x 4.6-mm (5-<math>\mu</math>m), Supelco LC-18 or equivalent?</p> <p style="padding-left: 40px;">(2) CN reverse phase HPLC column, 25-cm x 4.6-cm (5-<math>\mu</math>m), Supelco LC-CN or equivalent?</p> <p>e. If an "equivalent" column is in use, has its ability to generate data of acceptable accuracy and precision been demonstrated?</p> <p>f. Is the HPLC column temperature controlled? If not, is special care taken to ensure that temperature shifts do not cause peak misidentification?</p> <p>g. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p>		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
h. Has any instrument been modified in any way?  i. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?  j. Is a temperature controlled ultrasonic bath available?  k. Are backup apparatus available?		
Reagents:  a. Is reagent water used free from interferences at the MDL of target analytes?  b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Are "HPLC grade" or equivalent solvents used for explosives analysis?  d. Is sodium chloride stored in glass container?  e. Are all solvents stored in glass containers and transferred with all glass system?  f. Does the lab have calibration standards for all method specified target analytes?  g. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		

**CHART I-22**

**ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)**

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ITEM	YES	COMMENT
Sample Handling and Storage:  a. Are aqueous samples stored at 4°C, and extracted within seven days from collection and analyzed within 40 days from extraction?  b. Are soil samples stored at 4°C, and extracted within 14 days from collection and analyzed within 40 days from extraction?  c. Are all samples and sample extracts stored in the dark at 4°C?		
Instrument Calibration and Maintenance:  a. Is there a calibration protocol readily available to bench chemists?  b. Are calibration results kept in permanent logbooks?  c. Are solid analyte standards dried to constant weight in a vacuum desiccator in the dark prior to use?  d. Are stock standard solutions stored in refrigerator at 4°C in the dark and replaced after one year or sooner, if comparison with check standards indicates a problem?  e. Are intermediate standard solutions prepared in acetonitrile for both water and soil samples?  f. Are intermediate standard solutions stored in refrigerator at 4°C in the dark and replaced after six months or sooner, if comparison with check standards indicates a problem?		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
g. Are standards for low level methods and working standards prepared fresh on the day of calibration and stored in the dark?		
h. Is a 5 g/L calcium chloride solution added to each working standard?		
i. Is one of the calibration standards at a concentration near, but above, the MDL?		
j. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
k. Is an initial calibration performed with a minimum of five concentration levels for each target analyte?		
l. Does the initial calibration contain triplicate injections of each calibration standard?		
m. Is the response factor for each analyte taken as the slope of the best-fit linear regression line with correlation coefficient $\geq 0.995$ ?		
n. Is the calibration curve or factor verified with, at a minimum, a midpoint calibration standard in triplicate at the beginning of the day, singly at the midpoint of the run and after the last sample of the day, assuming a sample group of ten or less?		
o. Is an additional mid-level standard checked after each ten samples in the analytical batch?		

**CHART I-22**

**ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)**

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ITEM	YES	COMMENT
<p>p. Is a new calibration curve prepared for any target analyte when the response factors for the daily calibrations vary from the initial response factors by more than 15%?</p> <p>q. Is the retention time window established with three injections of two standard mixtures, (1) HMX, RDX, 135-TNB, 13-DNB, NB, 246-TNT, and 24-DNT, and (2) Tetryl, 26-DNT, 2-NT, 3-NT, and 4-NT, through the course of a 72-hour period?</p> <p>r. Is the retention time window checked on a quarterly basis or whenever a new HPLC column is installed?</p> <p>s. Is the retention time for each analyte in the daily mid-concentration standard used as the midpoint of the window for that day?</p>		
<p>Sample Preparation:</p> <p>a. Are process waste samples screened with the high-level method to determine if the low-level method (1-50 µg/L) is required?</p> <p>b. Is low-level method routinely used for most groundwater samples?</p> <p>c. Are soil samples dried in air at room temperature or colder to a constant weight without exposure to direct sunlight?</p> <p>d. Are dried soil samples ground and homogenized to pass a 30 mesh sieve?</p>		

CHART I-22

ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
<p>e. Are soil samples extracted in a cooled ultrasonic bath (&lt;30°C) for 18 hours?</p> <p>f. Is a salting-out procedure used for extraction and concentration of water samples?</p> <p>g. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Does the mobile phase consist of 50/50 (v/v) methanol/organic-free reagent water?</p> <p>b. Are peak heights used for quantitation of target analytes? (Peak height is recommended to improve the reproducibility of low level samples.)</p> <p>c. Are all positive measurements observed on the C-18 column confirmed with the CN column?</p>		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific MDL equal to or lower than the method specified MDL?</p>		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
<p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. To demonstrate that the lab can generate data of acceptable accuracy and precision, does the analyst perform the following operations?</p> <p>(1) Is an LCS prepared with standards independent from calibration standards analyzed for each batch?</p> <p>(2) Are replicate aliquots (at least four) of LCS analyzed, and average recovery and standard deviation of the recovery calculated for each target analyte using the four results to check the system performance?</p> <p>(3) If any individual standard deviation of recovery exceeds the method specified precision limits or any individual average recovery falls outside the method specified range for accuracy, is the analysis of actual samples halted until the system performance is back in control?</p> <p>g. Does the matrix spike solution contain at least one isomer of all target analytes?</p> <p>h. Does the lab routinely perform matrix spike and either one matrix duplicate or one matrix spike duplicate per batch of no more than 20 samples?</p>		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
<p>(1) If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory limit, is the spike at that regulatory limits or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(2) If the concentration of a specific analyte in a water sample is not checked against a limit, is the spike at the same concentration as the LCS or one to five times higher than the background concentration, whichever concentration would be higher?</p> <p>(3) If it is not possible to determine the background concentration, is the spike concentration</p> <ul style="list-style-type: none"> <li>- the regulatory limit, if any; or</li> <li>- the larger of either five times the expected background or LCS concentrations?</li> </ul> <p>(4) For other matrices, is the spike concentration at 20 times the estimated quantitation limit?</p> <p>(5) Is the percent recovery for each analyte in water samples checked with the method specified QC acceptance criteria?</p>		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
<p>(6) If the spike to background ratio is less than 5:1, does the lab use optional QC acceptance criteria calculated for the specific spike concentration?</p> <p>i. Does the lab use one or two analytes which are not expected to be presented in the sample as surrogates?</p> <p>j. Are the average percent recovery and standard deviation of percent recovery for each surrogate standard calculated when surrogate data from 25 to 30 samples for each matrix is available?</p> <p>k. Are control limits for each surrogate in a given matrix calculated based on the above data?</p> <p>l. At a minimum, are surrogate recovery limits updated annually on a matrix-by-matrix basis?</p> <p>m. Are corrective actions of reanalysis or reextraction/reanalysis taken if surrogate(s) for a sample are out of control limits?</p> <p>o. Are control charts for internal QC data plotted and available to operators?</p> <p>p. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		

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ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for explosives analysis?</p>		

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# CHART I-22

## ORGANIC ANALYSIS BY HPLC: EXPLOSIVES (8330)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for explosives analysis?		
Additional observations, comments, or problems:		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
<p>General:</p> <p>a. Are written SOPs available and adequate for sample preparation?</p> <p>b. Do these SOPs accurately reflect procedures in use?</p> <p>c. Are all sample preparations conducted in a hood?</p> <p>d. Are a group of samples (up to a maximum of 20) which behave similarly with respect to the procedures being employed and which are processed as a unit with the same method sequence and the same lots of reagents and with the reagents and with the manipulations manipulations common to each samples within the same time period or in continuous sequential time periods considered as a batch?</p> <p>e. Are the following lab internal QC samples prepared for each batch of samples?</p> <p>(1) Method blanks?</p> <p>(2) Matrix spikes?</p> <p>(3) Matrix spike duplicates?</p> <p>(4) Matrix duplicates?</p> <p>(5) Laboratory control samples?</p> <p>f. If the quantity of field samples is not sufficient for internal QC analyses, are blank spike/blank spike duplicate or duplicate laboratory control sample: analyzed?</p>		

**CHART I-23**

**SAMPLE PREPARATION FOR METAL ANALYSIS:**

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ITEM	YES	COMMENT
g. Are the rates of internal QC samples consistent with method requirements or, at a minimum, 5% per batch of no more than 20 samples with similar matrix, whichever is greater?		
h. Is the appropriateness of a particular preparation for a specific sample type determined by the completeness of extraction and by spike recoveries?		
i. Are logbooks for sample preparation used and well maintained?		
j. Are permanently bound notebooks with consecutively numbered pages used?		
k. Is a unique serial number clearly displayed on each notebook?		
l. Are critical times entered in logbooks?		
m. Are spiking solutions traceable to NIST or other reliable standards?		
n. Are spiking solutions labeled properly with date of preparation, composition, concentration, and identity of preparer?		
o. Have entries been made in permanent fashion and corrections made without obliterating original entries?		
p. Are corrections reviewed and initialed by a supervisor?		
q. Does the logbook of sample preparation contain the following information?		
(1) Date/time?		

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SAMPLE PREPARATION FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
(2) Sample ID number? (3) Sample preparer? (4) Matrix noted? (5) Spiking standards? (6) Pretreatment? (7) Volume/weight of sample? (8) Final volume? (9) Preparation methods?		
Acid Digestion of Mercury Samples for CVAA: a. Are mercury in liquid samples prepared according to Method 7470? b. Are mercury in solid or semisolid samples prepared according to Method 7471? c. Are all blanks, spiked samples, and laboratory control samples carried through the same digestion process?		
Acid Digestion of Aqueous Samples for FLAA and ICP (Method 3005A): a. Is this digestion used to prepare surface and ground water samples for analysis of total recoverable metals and dissolved metals by FLAA and ICP? b. For dissolved metals, is the samples filtered through a 0.5- $\mu$ m filter at the time of collection, prior to acidification with nitric acid?		

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SAMPLE PREPARATION FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
<p>c. Are samples digested with a mixture of concentrated nitric acid and hydrochloric acid?</p> <p>d. Is the sample heated at 90 to 95°C to avoid boiling and loss of antimony?</p> <p>e. Is filtration of digestate done only there is concern of insoluble materials may clog the nebulizer?</p> <p>f. Are the reagent water, nitric acid, and hydrochloric acid monitored to determine levels of impurities?</p> <p>g. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process?</p>		
<p>Acid Digestion of Aqueous and Extract Samples for FLAA and ICP (Method 3010A):</p> <p>a. Is this digestion used to prepare aqueous samples, TCLP extracts, and wastes that contain suspended solid for analysis of total metals by FLAA and ICP?</p> <p>b. Are samples digested with concentrated nitric acid?</p> <p>c. After the digestion is complete, is the sample warmed with 1:1 hydrochloric acid to dissolve any precipitate or residue?</p> <p>d. Is filtration of digestate done only there is concern of insoluble materials may clog the nebulizer?</p>		

CHART I-23

SAMPLE PREPARATION FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
<p>e. Are the reagent water, nitric acid, and hydrochloric acid monitored to determine levels of impurities?</p> <p>f. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process?</p> <p>g. Is the use of this digestion method avoided when samples are to be analyzed by the GFAA technique?</p>		
<p>Acid Digestion of Aqueous and Extract Samples by GFAA (Method 3020A):</p> <p>a. Is this digestion used to prepare aqueous samples, TCLP extracts, and wastes that contain suspended solid for analysis of total metals by GFAA?</p> <p>b. Is the digestion based on the use of nitric acid alone?</p> <p>c. Are the reagent water and nitric acid monitored to determine levels of impurities?</p> <p>d. Are all method blanks, spiked samples and laboratory control samples carried through the same digestion process?</p> <p>e. Are aqueous samples of arsenic and selenium prepared according to Methods 7060 and 7740, respectively?</p>		
<p>Acid Digestion of Oils, Greases, or Waxes ICP (Method 3040):</p> <p>a. Is the use of this preparation method limited to samples being analyzed only for Sb, Be, Cd, Cr, Cu, Fe, Mn, Ni, and V?</p>		

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**SAMPLE PREPARATION FOR METAL ANALYSIS:**

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ITEM	YES	COMMENT
b. Is xylene or methyl isobutyl ketone used as the solvent in this method?  c. Are organic metallic standards used?  d. Are method blanks (e.g., Conostan base oil or mineral oil plus reagents) spike samples, and laboratory control samples carried through the same preparation and analytical processes?  e. Are samples and standards diluted as closely as possible to the time of analysis?  f. Is the method of standard additions employed for all samples?  g. Is background correction employed to account for additive interferences?		
Acid Digestion of Sediments, Sludges, and Soils (Method 3050A):  a. Are nonaqueous samples refrigerated upon receipt and analyzed as soon as possible?  b. Are the samples mixed thoroughly to achieve homogeneity prior to digestion?  c. Is the initial phase of the digestion accomplished with nitric acid and hydrogen peroxide?  d. Is hydrochloric acid used as the final reflux acid for (1) the ICP analysis of As and Se, and (2) the FLAA and ICP analyses of Al, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Mo, Pb, Ni, K, Na, Tl, V, and Zn?		

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SAMPLE PREPARATION FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
<p>e. Is the use of hydrochloric acid avoided and nitric acid employed as the final dilution acid for GFAA analysis of As, Be, Cd, Cr, Co, Pb, Mo, Se, Tl, and V?</p> <p>f. Are the reagent water, nitric acid, hydrochloric acid, and hydrogen peroxide monitored to determine levels of impurities?</p> <p>g. Are all method blanks, spiked samples, and laboratory control samples carried through the same digestion process?</p> <p>h. Is the method of standard additions employed whenever a new sample matrix is analyzed?</p>		
<p>Alkaline Digestion for Hexavalent Chromium (Method 3060):</p> <p>a. Are samples digested with 3% sodium carbonate and 2% sodium hydroxide solution?</p> <p>b. Is the digestion solution stored in a tightly capped polyethylene bottle and prepared fresh monthly?</p> <p>c. Are the sample and digestate stored at 4°C until analyzed?</p> <p>d. Are all positive samples spiked with Cr (VI) to double the concentration found in the original aliquot, but with the increase no less than 0.10 mg/g?</p> <p>e. If spike recovery is not within 85% and 115%, is an interference regarded to be presented and the results invalid?</p>		

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**SAMPLE PREPARATION FOR METAL ANALYSIS:**

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ITEM
Additional observations, comments, or problems:

CHART I-24

GENERAL QA/QC FOR METAL ANALYSIS:

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ITEM	YES	COMMENT
<p>Atomic Absorption Spectrophotometer:</p> <ul style="list-style-type: none"> <li>a. Are fuels and oxidants commercial grade?</li> <li>b. Is there a filter moisture trap between the air source and the spectrometer?</li> <li>c. Is nitrous oxide reagent grade?</li> <li>d. Are flash-back arrestors and heaters in use where needed?</li> <li>e. Are all lamps dated when first put into use?</li> <li>f. Are lamps available for all elements analyzed?</li> <li>g. Does the lab have a Zeeman background correction system?</li> <li>h. Does the lab have a deuterium background correction system?</li> <li>i. Does the lab have a Smith-Hieftje background correction system?</li> </ul>		
<p>ICP-Atomic Emission Spectrometer:</p> <ul style="list-style-type: none"> <li>a. Is a background correction technique in use and documented according to sample matrix at least quarterly?</li> <li>b. Has the instrument detection limit and method detection limit for each element been established and documented at least semiannually?</li> <li>c. Where required, has the effect of high dissolved solids and/or acid concentration been controlled?</li> </ul>		

**CHART I-24**

**GENERAL QA/QC FOR METAL ANALYSIS:**

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ITEM	YES	COMMENT
d. Has salt buildup on the nebulizer been controlled?		
f. When a new matrix is encountered, is a serial dilution, spike addition, or an alternate method technique in use to eliminate potential interference?		
g. Is the spectrometer equipped with an argon gas supply?		
h. Are ultra high purity grade nitric acid, hydrochloric acid, and deionized or distilled water used for sample processing and preparation?		
Are manufacturer's operating manuals readily available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Are calibration results kept in permanent logbooks?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Is ICP calibration checked using a blank and the highest mixed calibration standard prior to sample analysis?		
Is ICP calibration verified every ten samples and at the end of the analytical run, using a calibration blank and a check standard?		
Does the result of check standard agree within $\pm 10\%$ of expected value?		

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**GENERAL QA/QC FOR METAL ANALYSIS:**

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ITEM	YES	COMMENT
Are interelement and background correction factors at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent?		
Does the result of interelement check sample agree within $\pm 20\%$ of expected value?		
Has the instrument been modified in any way?		
Are the instruments properly vented?		
Is an initial 5-point calibration run to check instrument linearity?		
Is the MDL for each element and matrix type determined every six months or whenever there is a significant change in background or instrument response?		
Is the linear calibration range determined for each element when there is significant change in instrument response and every six months for those elements that periodically approach their linear limits?		
Is a matrix spike run at a rate of 5% with each batch of samples?		
Is a corrective action taken if matrix spike recoveries exceed QC limits?		
Is an internal QC duplicate run at a rate of 5% with each batch of samples?		
Is a corrective action taken if the internal QC duplicate exceed QC limits?		
Is the method of standard addition in use where needed?		

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**CHART I-24**

**GENERAL QA/QC FOR METAL ANALYSIS:**

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ITEM
Additional observations, comments, or problems:

CHART I-25

METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
General:  a. Are written SOPs available and adequate for ICP sample preparation/analysis?  b. Do these SOPs accurately reflect procedures in use?  c. Are manufacturer's operating manuals readily available to bench chemists?  d. Are prenumbered, bound notebooks used for data entry?  e. Are all records written in indelible ink?  f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?  g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff:  a. Do bench chemists appear experienced with operation of an ICP system and knowledgeable in the correction of spectral, chemical, and physical interferences?  b. Are backup bench chemists available?  c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

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**CHART I-25**

**METAL ANALYSIS BY ICP: METALS (6010A)**

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ITEM	YES	COMMENT
Apparatus and Facilities:  a. Is working space adequate and clean?  b. Does the lab have a simultaneous multielement ICP?  c. Does the lab have a sequential multi-element ICP?  d. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?  e. Has any instrument been modified in any way?  f. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?  g. Are backup instruments available?  h. Are hoods used in sample preparation areas free of rust?		
Reagents:  a. Is reagent water used free from interferences at the MDLs of target analytes?  b. Is reagent grade water of at least 16 mega-ohm quality used for metal analysis?  c. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
<p>d. Are ultra-high purity chemicals or metals (99.99 to 99.999% pure) used for in-house preparation of standard stock solutions?</p> <p>e. Are all salts used for preparation of standard stock solutions dried for one hour at 105°C, unless otherwise specified?</p> <p>f. If standard stock solutions are purchased, are the concentrations of the analytes verified in-house?</p> <p>g. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem?</p> <p>h. Are calibration standards initially verified using check standards and monitored weekly for stability?</p> <p>i. Are silver standards limited to 2 mg/L and prevented from exposure to light?</p> <p>j. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?</p>		
<p>Sample Handling and Storage:</p> <p>a. Are aqueous samples preserved at <math>\text{pH} \leq 2</math> with nitric acid?</p> <p>b. Are solid samples stored at 4°C?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol readily available to bench chemists?</p>		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
b. Are calibration results kept in permanent logbooks?  c. Is the linearity of ICP calibration range established with a minimum of five levels of calibration standards?  d. Is an initial calibration performed with a minimum of three concentration levels for each target analyte?  e. Does the lab empirically establish the detection limits, sensitivity, and optimum ranges of the metals for each model of spectrometer and type of matrices?  f. Are multiple exposures conducted to secure a reliable average reading for each solution?  g. Is one of the calibration standards at a concentration near, but above, the MDL?  h. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?  i. Are all mixed calibration standard solutions scanned with a sequential spectrometer to verify the absence of interelement spectral interference?		
Sample Preparation:  a. Is an appropriate sample preparation method, Methods 3005A, 3010A, 3020A, 3040, or 3050A, used for sample digestion?		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
b. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?		
<p>Sample Analysis:</p> <p>a. Is an ICP allowed to become thermally stable before beginning calibration or analysis (usually requiring at least 30 minutes)?</p> <p>b. Are the average intensity of multiple exposures for both standardization and sample analysis used to reduce random error?</p> <p>c. Before beginning the sample run, is the highest mixed calibration standard reanalyzed to check if the deviation is within 5% from actual value?</p> <p>d. Is daily calibration checked with a mid-concentration standard at the beginning and the end of an analysis sequence?</p> <p>e. Is sufficient quantity of calibration blank solution used to flush the system for at least one minute before the analysis of each standard or sample?</p> <p>f. If a peak response exceeds the linear range of the system, is a dilution performed with calibration blank solution on a second aliquot of the sample that has been properly sealed and stored prior to use?</p> <p>g. Is an alternate less sensitive spectral line used only when all QC data are already established?</p>		

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**CHART I-25**

**METAL ANALYSIS BY ICP: METALS (6010A)**

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ITEM	YES	COMMENT
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Are lab specific IDL and MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific IDL or MDL equal to or lower than the method specified IDL or MDL, respectively?</p> <p>e. Is a calibration blank used in establishing the calibration curve?</p> <p>(A calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples.)</p> <p>f. Is a minimum of one method blank per sample batch used to determine any memory effects or possible contaminations resulting from varying amounts of the acids used in the sample processing?</p> <p>(A method blank must contain all reagents in the same volumes as used in the processing of the samples and must be carried through the complete procedure and contain the same acid in the final solution as the sample solution used for analysis.)</p>		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
<p>g. When a new or unusual sample matrix is encountered, are the following series of tests conducted to check interferences?</p> <p>(1) Serial Dilution: If the analyte concentration is minimally 50 times higher than the IDL, is a fivefold dilution analyzed and compared with the original determinations within 10%?</p> <p>(2) Post Digestion Spike Addition: Is an analyte spike added to a prepared sample, or its dilution to produce a minimum level of ten times and a maximum of 100 times of the IDL recovered to within 25% of the known value?</p> <p>h. If the above tests fail and interferences are suspected, are corrective actions such as use of a standard-addition analysis, computerized compensation, an alternate wavelength, or comparison with an alternative method used?</p> <p>i. Is the ICP calibration checked using a calibration blank and two appropriate standards?</p> <p>j. Is ICP calibration verified every ten samples and at the end of the analytical run, using a calibration blank and a check standard?</p> <p>k. Is the check standard prepared with reference materials independent of calibration standards analyzed for each batch?</p>		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
<p>l. Does the result of the calibration blank agree within 30 of mean blank value? If not, are the blank analysis repeated twice and the results averaged and checked against the 30 of the background mean?</p> <p>m. If the check standard is not within 10% of the expected value or the average background is not within 30, is the analysis terminated, the problem corrected, the instrument recalibrated, and the analysis of previous ten samples repeated?</p> <p>n. Are the interelement and background correction factors verified at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent? (The results should be within 20% of true values.)</p> <p>o. To demonstrate that a lab can generate data of acceptable accuracy and precision, does the lab routinely perform matrix spike, matrix spike duplicate, and matrix duplicate per batch of no more than 20 samples?</p> <p>p. Is a control limit of <math>\pm 20\%</math> RPD used for sample values greater than ten times the IDL?</p> <p>q. Is the control limit for spike duplicate sample within 20% of the actual value?</p> <p>r. Are control charts for internal QC data plotted and available to bench chemists?</p>		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM	YES	COMMENT
<p>s. Are control limits for internal quality control empirically established and updated on a regular basis?</p> <p>t. Are all results reported with up to three significant figures?</p>		
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		

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**METAL ANALYSIS BY ICP: METALS (6010A)**

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ITEM	YES	COMMENT
Overall Evaluation:  a. Does the lab have sound technical capability for ICP analysis?  b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for ICP analysis?		

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METAL ANALYSIS BY ICP: METALS (6010A)

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ITEM
Additional observations, comments, or problems:

## CHART I-26

## METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPS available and adequate for AA sample preparation and analysis?</li> <li>b. Do these SOPS accurately reflect procedures in use?</li> <li>c. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>d. Are prenumbered, bound notebooks used for data entry?</li> <li>e. Are all records written in indelible ink?</li> <li>f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?</li> <li>g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear experienced with operation of an AA system and knowledgeable in the correction of spectral, chemical, and physical interferences?</li> <li>b. Are backup bench chemists available?</li> <li>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</li> </ul>		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
<p>Apparatus and Facilities:</p> <p>a. Is working space adequate and clean?</p> <p>b. Does the lab have in-house capability for metal analysis by FLAA, GFAA, CVAA, and HGAA?</p> <p>c. Does the lab have a Zeeman background correction system for GFAA?</p> <p>d. Does the lab have other background correction systems for GFAA (e.g., deuterium and/or Smith-Hieftje)?</p> <p>e. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?</p> <p>f. Has any instrument been modified in any way?</p> <p>g. Are analytical balance (0.0001 g) and top loading balance (0.01 g) available?</p> <p>h. Are backup instruments available?</p> <p>i. Are all glassware, polypropylene/ or Teflon containers, including sample bottles and flasks, washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and reagent water?</p> <p>j. Are pipet tips acid soaked with 1:5 HNO<sub>3</sub> and rinsed thoroughly with tap and deionized water (Type II ASTM D1193)?</p>		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
Reagents:  a. Is reagent water used free from interferents at the MDLs of target analytes?  b. Is reagent grade water of at least 16 mega-ohm quality used for metal analysis?  c. Do reagent grade chemicals used conform to the-specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  d. Are all reagents analyzed to prove that all constituents are below the MDLs?  e. Are spectrograde hydrochloric and nitric acids certified for AA analysis used for metal analysis?  f. Are redistilled nitric or hydrochloric acids used for preparation of stock standard metal solutions?  g. Are sulfuric or phosphoric acids avoided for standard preparation?  h. If standard stock solutions are prepared in-house, are all salts dried for one hour at 105°C, unless otherwise specified?  i. If standard stock solutions are purchased, are the concentrations of the analytes verified in-house?		

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METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
j. Are stock standards replaced after one year, or sooner if comparison with check standards indicates a problem? k. Are calibration standards initially verified using check standards and monitored weekly for stability? l. Is the check standard prepared with reference materials independent of calibration standards analyzed for each batch? m. Are silver standards limited to 2 mg/L and prevented from exposure to light? n. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified? o. Is the acetylene tank grounded, safely strapped, and >100 psi?		
Sample Handling and Storage: a. Are aqueous samples preserved at pH $\leq$ 2 with nitric acid? b. Are solid samples stored at 4°C?		
Instrument Calibration and Maintenance: a. Is there a calibration protocol readily available to bench chemists? b. Are calibration results kept in permanent logbooks?		

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**CHART I-26**

**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
c. Is a calibration curve prepared each day with a minimum of three (except five for mercury) concentration levels for each analyte?		
d. Are equal amounts of permanganate reagents added to mercury calibration standards and blanks?		
e. Is a calibration curve made for every hour of continuous sample analysis of mercury, arsenic, or selenium by CVAA or GFAA, respectively?		
f. Are freshly prepared calibration standards used each time a batch of samples is analyzed?		
g. Are the absorbance readings of calibration standards within 0.0 and 0.7?		
h. Are multiple exposures conducted to secure a reliable average reading for each solution?		
i. Is one of the calibration standards at a concentration near, but above, the MDL?		
j. Do concentrations of other standards cover the expected concentration ranges of real samples or define the working range of the detector?		
Sample Preparation:		
a. Is an appropriate sample preparation method, Methods 3005A, 3010A, 3020A, 3040, or 3050A, used for sample digestion?		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
<p>b. Are the digestion procedures in Section 7.1 of Methods 7060 and 7740 used for preparation of aqueous arsenic and selenium samples, respectively?</p> <p>c. Are the digestion procedures in Section 7.0 of Methods 7470 and 7471 used for preparation of aqueous and solid mercury samples, respectively?</p> <p>d. For seawater, brines, and industrial effluents high in chlorides, are additional hydroxylamine sulfate and permanganate reagents (25 mL) used to prevent chlorine interference?</p> <p>e. Are soil samples dried at ambient temperature, ground, and sieved, prior to subsampling?</p> <p>f. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p>		
<p>Sample Analysis:</p> <p>a. Are the instructions provided by the manufacturer followed for each AA?</p> <p>b. After choosing the proper lamp for analysis, is the lamp allowed to warm up for a minimum of 15 minutes, unless operated in a double-beam mode?</p> <p>c. Is an instrument blank run and the instrument zeroed?</p> <p>d. Is a lanthanum solution added to samples that are to be analyze for calcium and magnesium?</p>		

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**CHART I-26**

**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
e. Is a calcium solution added to samples that are to be analyzed for iron and magnesium?		
f. Is a potassium chloride solution added to samples before atomization in the determination of aluminum, barium, and titanium?		
g. Is an aluminum nitrate solution added to samples before atomization in the determination of molybdenum and vanadium?		
h. Is a cyanogen iodide solution added to samples that are to be analyzed for silver?		
i. Is an unused cyanogen iodide solution discarded after two weeks and fresh solution prepared?		
j. Is a cyanogen iodide solution kept away from any acid solution?		
k. If a nitrous oxide/acetylene flame is used, is the nitrous oxide cylinder fitted with a non-freezable regulator or is a heating coil wrapped around an ordinary regulator?		
l. After a nitrous oxide/acetylene flame has been ignited, is the burner allowed to come to thermal equilibrium before the analysis is begun?		
m. Are the average intensity of multiple exposures for both standardization and sample analysis used to reduce random error?		

**CHART I-26**

**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
<p>n. If the concentration found is greater than the highest standard, is the sample diluted in the same acid matrix and reanalyzed?</p> <p>o. Is same injection volumes used for samples and standards?</p> <p>p. Is a magnesium perchlorate drying tube or a small 60-W light bulb used to prevent condensation of moisture inside a mercury absorption cell?</p>		
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review carried out within the lab prior to data release?</p> <p>c. Are lab specific IDL and MDL empirically established and updated on a semiannually basis?</p> <p>d. Is the lab specific IDL or MDL equal to or lower than the method specified IDL or MDL, respectively?</p> <p>e. Is a calibration curve prepared each day with a minimum of a calibration blank and three standards?</p> <p>f. Is the calibration curve verified with at least a calibration blank and a mid-range check standard made from reference material or other independent standard material? (The check standard must be within 10% of its value for the curve to be considered valid.)</p>		

**CHART I-26**

**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
<p>g. If more than ten samples per day are analyzed, is the calibration curve verified with a mid-range calibration standard or check standard after every ten samples? (This sample value must be within 20% of the true value, or the previous ten samples need to be reanalyzed.)</p> <p>h. For mercury, arsenic, or selenium analysis by CVAA or GFAA, is the calibration curve verified with a mid-range, independently prepared check check standard every 15 samples?</p> <p>i. For mercury, arsenic, or selenium analysis by CVAA or GFAA, are the samples diluted if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve?</p> <p>j. Are the following interference tests conducted for each analytical batch?</p> <p>(1) Dilution Test: Select one typical sample with concentration of analytes at <math>\geq 25</math> times of the MDL. Dilute the sample by a minimum of fivefold and analyze. If the concentrations between the diluted and the undiluted are within 10%, the absence of interferences can be assumed and samples may be analyzed without using method of standard additions.</p>		

CHART I-26

METAL ANALYSIS BY AA: METALS (7000s)

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ITEM	YES	COMMENT
<p>(2) Recovery Test: If all samples in the batch are below ten times the MDL or the Dilution Test fails, a spiked sample should be analyzed. Add a known amount of analyte to bring the concentration to two to five times the original concentration or to 20 times of the MDL if all analytes in the batch are below MDL. The spike recovery should be within 15%, otherwise the method of standard additions shall be used for all samples in the batch.</p> <p>k. To demonstrate that a lab can generate data of acceptable accuracy and precision, does the lab routinely perform matrix spike, matrix spike duplicate, and matrix duplicate at a minimum rate of 5% or one per batch, whichever is greater?</p> <p>l. Is a control limit of <math>\pm 20\%</math> RPD used for sample values greater than ten times the IDL?</p> <p>m. Is the control limit for spike duplicate sample within 20% of the actual value?</p> <p>n. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>o. Are control limits for internal quality control empirically established and updated on a regular basis?</p> <p>p. Are all results reported with up to three significant figures?</p>		

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**CHART I-26**

**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
Data Package:  a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)  b. Does the data package contain all method required QC data and meet the USACE contract requirements?  c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal:  a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?  b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?		
Overall Evaluation:  a. Does the lab have sound technical capability for AA analysis?		

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**METAL ANALYSIS BY AA: METALS (7000s)**

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for AA analysis?		
Additional observations, comments, or problems:		

**CHART I-27**

**GENERAL QA/QC FOR CLASSICAL ANALYSIS:**

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ITEM	YES	COMMENT
Is the wavelength accuracy and repeatability of all spectrophotometers checked at several wavelengths for each batch of samples?		
Is photometric accuracy and repeatability checked and documented with NIST-traceable standards?		
Is acid washed glassware retained for phosphorus analyses only?		
Is ammonia free water used in preparation of standards and samples for nitrogen analyses?		
Are manufacturer's operating manuals available to bench chemists?		
Is there a calibration protocol available to bench chemists?		
Are calibration results kept in permanent logbooks?		
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
Has any instrument been modified in any way?		
Is a minimum of 4-point calibration used?		
Are continuing calibration checks done on a regular basis?		
Is the MDL for each analyte and matrix type determined every six months or whenever there is a significant change in background or instrument response?		

CHART I-27

GENERAL QA/QC FOR CLASSICAL ANALYSIS:

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ITEM	YES	COMMENT
Is the linear calibration range determined for each analyte when there is significant change in instrument response and every six months for those analytes that periodically approach their linear limits?		
Are internal QC samples run per method requirements?		
Is a method blank run for each batch?		
Additional observations, comments, or problems:		

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**CHART I-28**

**CLASSICAL ANALYSIS: COMMON ANIONS (300s)**

Page 1 of 7

ITEM	YES	COMMENT
Does the lab use an ion chromatography (IC) to analyze chloride, fluoride, nitrate, nitrite, ortho-phosphate, and sulfate?		
Does the IC system have appropriate anion guard column, separator column, suppressor column, and conductivity detector?		
Is the maximum loading to a separator column kept below 400 µg/L to avoid column overloading and nonlinear response?		
Are nitrate, nitrite, ortho-phosphate, and sulfate samples stored at 4°C?		
Are the lab's holding times for nitrate, nitrite, and ortho-phosphate by IC method 48 hours from sampling to analysis? (28 days for chloride, fluoride, and sulfate.)		
Is the eluent solution made of sodium bicarbonate (0.003 M) and sodium carbonate (0.0024 M)?		
Is the regeneration solution made of sulfuric acid (0.025 N)?		
Is a filtration conducted on samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns?		
Is a reagent water analyzed before processing any standards or samples to demonstrate that all glassware and reagent interferences are under control?		
Is a reagent blank processed each time there is a change in reagents?		

CHART I-28

CLASSICAL ANALYSIS: COMMON ANIONS (300s)

Page 2 of 7

ITEM	YES	COMMENT
Are calibration standards prepared from sodium chloride, sodium fluoride, sodium nitrate, sodium nitrite, potassium sulfate, and potassium dihydrogen phosphate dried at 105°C for 30 minutes?		
Are stock standards stored at 4°C?		
Are working standards prepared at a minimum on a weekly basis, except those for nitrite and phosphate which should be prepared fresh daily?		
Is a minimum of three concentration levels and a blank used for calibration of each analyte of interest?		
Is one of the calibration standards near, but above, the MDL?		
Is the injection loop flush thoroughly using each new standard or sample?		
Is the same size of injection loop used for standards and samples?		
Unless the attenuator range settings are proven to be linear, is each setting calibrated individually?		
If the working range exceeds the linear range of the system, is a sufficient number of standards analyzed to allow an accurate calibration curve to be established?		
Is the water dip or negative peak that elutes near and interferes with fluoride peak eliminated by the addition of concentrated eluent to each standard and sample?		

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**CHART I-28**

**CLASSICAL ANALYSIS: COMMON ANIONS (300s)**

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ITEM	YES	COMMENT
Are the retention times of each analyte documented during the calibration? (Retention time is inversely proportional concentration.)		
Is the working calibration curve verified on each working day, or when the anion eluent is changed, and after every 20 samples?		
If the response or retention time for any analyte varies from the expected values by more than $\pm 10\%$ , is the test repeated with fresh calibration standards?		
If the results are still more than $\pm 10\%$ , is an entirely new calibration curve prepared for that analyte?		
Is the width of retention time window determined based upon three times of standard deviation of measurements of actual retention time variations over the course of a day?		
If the response of a peak exceeds the working range of the system, is the sample diluted with reagent water and reanalyzed?		
Is an initial demonstration of laboratory capability conducted with a minimum of four LCS?		
Is a continuing check on laboratory performance conducted with spiked samples at a minimum rate of 10% of all samples?		
Are LCS, lab duplicates, and other QC check samples routinely analyzed for each sample batch?		

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CLASSICAL ANALYSIS: COMMON ANIONS (300s)

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ITEM	YES	COMMENT
Are method performance criteria empirically determined for each spike concentration of analyte being measured?		
Does the lab develop and maintain separate accuracy statements, $\%R \pm \sigma$ , for water and wastewater samples? (The average percent recovery, $\%R$ , and the standard deviation of the percent recovery, $\sigma$ , are developed by analyses of four aliquots of water and wastewater.)		
Is a confirmatory technique such as sample dilution and spiking used to confirm anion identification?		
<p>Fluoride:</p> <p>a. Distillation:</p> <p>(1) Before a sample is run, is the distillation apparatus flushed by distilling the sulfuric acid-distilled water mixture until the temperature reaches 180°C?</p> <p>(2) Is the sample and acid-water mixture distilled until the flask temperature reaches 180°C?</p> <p>(3) Is the heating the contents of the distilling flask above 180°C avoided?</p> <p>(4) Are all water and wastewater sample distilled?</p> <p>b. Calorimetric-SPADNS:</p> <p>(1) If residual chlorine is present, is it removed with sodium arsenite solution?</p>		

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CLASSICAL ANALYSIS: COMMON ANIONS (300s)

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ITEM	YES	COMMENT
(2) Are all samples (including potable water) subjected to preliminary distillation?		
(3) Are standards prepared in the range of 0 to 1.40 mg/L?		
(4) Are samples and standards at the same temperature for color development?		
(5) Is color development carried out with SPADNS solution and zirconyl-acid reagent (or, alternatively acid-zirconyl-SPADNS reagent)?		
(6) Is the absorbance of samples and standards read at 570 nm?		
(7) Is a standard curve drawn based on the absorbance of the standards?		
(8) Are the fluoride concentrations of the samples read directly from the curve without extrapolation?		
(9) Are standard curves retained as part of the record?		
c. Potentiometric Ion Selective Electrode:		
(1) Is a series of fluoride standards covering the range of 0 to 2.0 mg/L fluoride prepared?		
(2) Is an equal volume of total ionic strength adjustment buffer mixed with the sample or standard to be measured?		
(3) Are samples and standards measured at room temperature?		

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CLASSICAL ANALYSIS: COMMON ANIONS (300s)

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ITEM	YES	COMMENT
(4) When a measurement is made, are the electrodes allowed to remain in the solution for three minutes (or longer if necessary) before a reading is made?		
(5) When an electrometer is used, is a standard curve prepared on semi-logarithmic graph paper with the fluoride concentration in mg/L on the log axis and the electrode potential developed in the standard on the linear axis?		
(6) Are the samples diluted and remeasured if they fall outside the working range of the standard curve?		
(7) Is a 1.00 mg/L fluoride standard read after each known sample and each standard?		
(8) If a selective-ion meter is used, is it calibrated in accordance with the manufacturer's instruction?		
(9) Are all standard curves and calibration data retained as part of the record?		

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**CHART I-28**

**CLASSICAL ANALYSIS: COMMON ANIONS (300s)**

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ITEM
Additional observations, comments, or problems:

**CHART I-29**

**CLASSICAL ANALYSIS: OIL AND GREASE (413.1)**

Page 1 of 2

ITEM	YES	COMMENT
Are samples collected in glass containers?		
Are samples preserved at the time of collection by adjusting the pH to two or less with hydrochloric acid or sulfuric acid and cooling to 4°C?		
Are samples analyzed within 28 days of collection?		
Are the samples at a pH of two or less when the analysis is begun?		
Is the sample level marked on the sample container for later determination of sample volume?		
Is the entire sample transferred to a separator funnel?		
Is the sample bottle carefully rinsed with fluorocarbon 113 for two minutes and the layers allow to separate?		
Is the solvent layer drained through a funnel containing solvent moistened filter paper and (if necessary) anhydrous sodium sulfate into clean tared distilling flask?		
Is the extraction repeated twice more and the extracts combined in the distilling flask?		
Is the solvent distilled from the distilling flask using a 70°C water bath as a source of heat?		
After the distillation is completed, is the distilling flask swept out with air by inserting a glass tube connected to a vacuum source?		

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CHART I-29

CLASSICAL ANALYSIS: OIL AND GREASE (413.1)

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ITEM	YES	COMMENT
Is the flask wiped clean and dry on the outside, cooled in a desiccator for 30 minutes, and then weighted?		
Is a solvent blank run with each set of samples?		
Quality Control Requirements: <ul style="list-style-type: none"> <li>a. Is a laboratory blank analyzed daily or with each batch of sample run?</li> <li>b. Is a reference standard analyzed with every tenth sample?</li> <li>c. Is a spiked sample analyzed with every 20th sample?</li> <li>d. Are duplicate analyses performed on a minimum of 10% of all positive samples?</li> </ul>		
Additional observations, comments, or problems:		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

Page 1 of 9

ITEM	YES	COMMENT
<p>General:</p> <ul style="list-style-type: none"> <li>a. Are written SOPs available and adequate for TRPH sample preparation/analysis?</li> <li>b. Do these SOPs accurately reflect procedures in use?</li> <li>c. Are manufacturer's operating manuals readily available to bench chemists?</li> <li>d. Are prenumbered, bound notebooks used for data entry?</li> <li>e. Are notebooks reviewed and initialed by supervisors on a regular basis?</li> <li>d. Is an error crossed out with a line and correction entered, dated, and initialed?</li> </ul>		
<p>Technical Staff:</p> <ul style="list-style-type: none"> <li>a. Do bench chemists appear knowledgeable and experienced in TRPH analysis?</li> <li>b. Are backup bench chemists available?</li> <li>c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?</li> </ul>		
<p>Apparatus and Facilities:</p> <ul style="list-style-type: none"> <li>a. Is working space adequate and clean?</li> <li>b. Are enough sets of separator funnels (2,000 mL with Teflon stopcock) and Soxhlet extractors available for simultaneous extraction of all batch samples?</li> </ul>		

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**CHART I-30**

**CLASSICAL ANALYSIS: TRPH (418.1)**

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ITEM	YES	COMMENT
c. Is a hood available for sample preparation?		
d. Are IR spectrophotometers suitable for measurements around 2930 cm <sup>-1</sup> ?		
e. Does lab have sodium chloride or IR grade optical cells of 1-cm, 5-cm, and 10-cm pathlength?		
f. Are backup apparatuses available?		
Reagents:  a. Is reagent water used free from interferents at the method detection limits of target analytes?  b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?  c. Is magnesium sulfate monohydrate prepared by drying the heptahydrate salt in an oven at 150°C overnight?  d. Is granular, anhydrous sodium sulfate purified by heating at 400°C for four hours, or by precleaning with Freon-113 (1,1,2-trichloro-1,2,2-trifluoroethane)?  e. Is silica gel, 60-200 mesh, containing 1-2% water as defined by residue test at 130°C available? (Dried at 110°C for 24 hours and stored in a tightly sealed container.)		

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CLASSICAL ANALYSIS: TRPH (418.1)

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ITEM	YES	COMMENT
f. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
<p>Sample Handling and Storage:</p> <p>a. Is the pH of aqueous and sludge samples checked and adjusted to &lt;2 during sample log-in?</p> <p>b. Are aqueous and sludge samples stored at 4°C and analyzed within 28 days?</p> <p>c. Are soil samples stored at 4°C and analyzed with minimum delay upon receipt in the lab?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol available to the bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Are IR spectrophotometric accuracy and repeatability checked and documented with NIST-traceable standards?</p> <p>d. Are the materials of interest, if available, or the same type of petroleum fraction, if it is known and original sample is unavailable, used for preparation of calibration standards?</p>		

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**CHART I-30**

**CLASSICAL ANALYSIS: TRPH (418.1)**

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ITEM	YES	COMMENT
e. Does the lab normally attempt to determine the petroleum fraction type for unknowns prior to instrument calibration? (Reference oil is to be used as a last resort for unknowns, as it generates low values for diesel, kerosene, and other known petroleum hydrocarbon types.)		
f. Does reference oil contain a mixture of n-hexadecane, isooctane, and chlorobenzene in the appropriate proportions? (i.e., 15.0 mL + 15.0 mL + 10.0 mL)		
g. Is Freon-113, b.p. 48°C, used for standard and sample preparation?		
h. Is a minimum of a four-point calibration curve (a blank plus three standards) prepared for calibration?		
i. Do working ranges and cell pathlengths comply with method requirements?		
j. Is a calibration plot prepared for absorbance versus mg petroleum hydrocarbons in 100 mL solution?		
k. Are standards scanned from 3200 $\text{cm}^{-1}$ to 2700 $\text{cm}^{-1}$ with solvent in the reference and results recorded on absorbance paper?		
l. Are absorbance of standards measured by constructing a base line over the scan range and measuring absorbance of the peak maximum at 2930 $\text{cm}^{-1}$ and subtracting absorbance at that point?		
m. Are continuing calibration checks done on a regular basis for each batch of samples?		

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CLASSICAL ANALYSIS: TRPH (418.1)

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ITEM	YES	COMMENT
n. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		
<p>Sample Preparation:</p> <p>a. For aqueous samples, are the sample bottles marked at the water meniscus for later determination of sample volume?</p> <p>b. Is the entire aqueous sample consumed for analysis and no analysis performed on aliquots of samples?</p> <p>c. Is the pH value of aqueous samples checked and adjusted to <math>\leq 2</math> prior to extraction?</p> <p>d. Are sample bottle, tip of separator funnel, filter paper, and funnel rinsed with solvent and the rinsate combined with extract?</p> <p>e. Is the aqueous sample sequentially extracted with three 30 mL portion of fresh Freon-113?</p> <p>f. Is sodium sulfate, anhydrous crystal, used when emulsion occurs?</p> <p>g. Is the percent solid of solid samples determined by drying overnight at 105°C in a vented drying oven?</p> <p>h. Are sludge samples acidified to a pH of two and dried with magnesium sulfate monohydrate?</p> <p>i. Are sediment/soil samples decanted and dried with anhydrous sodium sulfate?</p>		

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**CHART I-30**

**CLASSICAL ANALYSIS: TRPH (418.1)**

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ITEM	YES	COMMENT
j. For solid samples, is Soxhlet method (Method 9071, steps 7.1 thru 7.11), instead of sonication method, used for sample extraction?		
k. Is Soxhlet extraction conducted at a rate of 20 cycles per hour for four hours?		
l. Is the water bath kept at 70°C?		
m. Is extract filtered with grease-free cotton or glass wool that is cleaned with solvent?		
n. Is 3-g silica gel used to remove polar fatty matter by stirring the solution with a Teflon coated magnetic stirrer for a minimum of five minutes?		
o. Is the absorptive capacity of silica gel checked by repeating the silica gel treatment procedure?		
Sample Analysis:		
a. Are samples scanned from 3200 $\text{cm}^{-1}$ to 2700 $\text{cm}^{-1}$ with solvent in the reference beam and results recorded on absorbance paper?		
b. Is a straight baseline constructed over the scan range and subtracted from the peak maximum at 2930 $\text{cm}^{-1}$ ?		
c. If the absorbance exceeds 0.8, is a shorter pathlength cell or a diluted extract used?		
d. Does the lab strictly adhere to the method without any deviations?		

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CLASSICAL ANALYSIS: TRPH (418.1)

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ITEM	YES	COMMENT
<p>Quality Control:</p> <p>a. Are all QC data maintained and available for easy reference and inspection?</p> <p>b. Is a three-level data review conducted within the lab prior to data release?</p> <p>c. Is a lab specific MDL empirically established and updated on a semiannually basis?</p> <p>d. Does the lab specific MDL meet or exceed the method specified MDL?</p> <p>e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>f. Is calibration curve verified within <math>\pm 10\%</math> of an independent, mid-range check standard for each batch?</p> <p>g. Are duplicate analyses performed at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>h. Is one pair of matrix spike and matrix spike duplicate samples run at a minimum rate of 5% or one per batch, whichever is more frequent?</p> <p>i. Are control charts for internal QC data plotted and available to bench chemists?</p> <p>j. Are control limits for internal quality control empirically established and updated on a regular basis?</p>		

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CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

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ITEM	YES	COMMENT
Data Package:  a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)  b. Does the data package contain all method required QC data and meet the USACE contract requirements?  c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?		
Waste Disposal:  a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?  b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?  c. Does the lab recycle Freon-113?		
Overall Evaluation:  a. Does the lab have sound technical capability for TRPH analysis?		

CHART I-30

CLASSICAL ANALYSIS: TRPH (418.1)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____  c. Could the lab handle quick turnaround samples?  d. Overall, is the lab acceptable for TRPH analysis?		
Additional observations, comments, or problems:		

**CHART I-31**

**CLASSICAL ANALYSIS: CYANIDE (9010A)**

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ITEM	YES	COMMENT
General:  a. Are written SOPs available and adequate for cyanide sample preparation and analysis?  b. Do these SOPs accurately reflect procedures in use?  c. Are manufacturer's operating manuals readily available to bench chemists?  d. Are prenumbered, bound notebooks used for data entry?  e. Are all records written in indelible ink?  f. Are all errors corrected by drawing a single line through the error with corrections written adjacent to the error, so that it remains legible, and initialed and dated by the responsible individual?  g. Are notebooks reviewed, initialed, and dated by supervisors on a regular basis?		
Technical Staff:  a. Do bench chemists appear knowledgeable and experienced in cyanide analysis?  b. Are backup bench chemists available?  c. Are bench chemists' performance audited and approved prior to work without close supervision by a senior chemist?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

Page 2 of 10

ITEM	YES	COMMENT
<p>Apparatus and Facilities:</p> <p>a. Is working space adequate and clean?</p> <p>b. Are enough sets of reflux distillation apparatuses available for simultaneous distillation for all batch samples?</p> <p>c. Is a hood available for sample preparation?</p> <p>d. Are spectrophotometers suitable for measurements at 578 nm with a 1.0-cm cell or larger?</p> <p>e. Are backup apparatus available?</p>		
<p>Reagents:</p> <p>a. Is ASTM Type II water (ASTM D1193) monitored and used for analysis?</p> <p>b. Do reagent grade chemicals used conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available?</p> <p>c. Is KCN used for standard preparation in good physical condition?</p> <p>d. Is chloramine-T solution prepared fresh daily and refrigerated until ready to use?</p> <p>e. Is pyridine-barbituric acid reagent stored in a cool, dark place and discarded after six months (one month if stored at room temperature in the light) or upon formation of a precipitate?</p>		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
f. Are all reagents and standards labeled, dated, initialed, and documented such that composition and expiration date can be verified?		
<p>Sample Handling and Storage:</p> <p>a. Are the pH values of aqueous samples checked and adjusted to <math>\geq 12</math> in a hood during log-in?</p> <p>b. If aqueous samples are not run immediately, are oxidizing agents, such as chlorine, in the samples checked with acidified KI-starch paper and preserved with ascorbic acid during sample log-in?</p> <p>c. Are samples stored at 4°C and prepared within 14 days?</p>		
<p>Instrument Calibration and Maintenance:</p> <p>a. Is there a calibration protocol available to the bench chemists?</p> <p>b. Are calibration results kept in permanent logbooks?</p> <p>c. Are photometric accuracy and repeatability checked and documented with NIST-traceable standards?</p> <p>d. Are calibration standards traceable to NIST or other reliable standards?</p> <p>e. Is a minimum of one 7-point calibration curve (a blank plus six standards) prepared for calibration?</p>		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
f. Is method blank, consisting of sodium hydroxide dilution solution (1.25 N) and all reagents, used to adjust the photometer zero?		
g. Is a calibration curve ranging from 20 to 400 µg/L prepared?		
h. Are the cyanide standards prepared fresh daily and kept in glass-stoppered bottles?		
i. Are all calibration standards prepared with sodium hydroxide dilution solution for all dilution?		
j. Is a calibration curve prepared covering the range of the method by plotting absorbance of standards against cyanide concentrations (0-1.0 mg/L?)		
k. For samples without sulfide, is a minimum of two standards (a high and low) distilled and compared with similar values on the curve to test the distillation technique?		
l. Do the distilled standards agree within ±10% of the undistilled standards?		
m. For samples with sulfide, are all standards distilled in the same manner as the samples?		
n. Are continuing calibration checks done on a regular basis?		
o. Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
p. Has any instrument been modified in any way?		
Sample Preparation:  a. Is pretreatment for cyanides amenable to chlorination performed in a hood to avoid the very toxic gas cyanogen chloride and under amber light to avoid false positive from $K_3[Fe(CN)_6]$ decomposed by UV light?  b. During the chlorination procedure, is the pH maintained between 11 and 12, and residual chlorine checked and maintained for one hour while the samples are agitated by magnetic stirring bars?  c. After chlorination, is excess reducing agent, ascorbic acid or sodium arsenite, added to remove chlorine?  d. Are all samples distilled before cyanide determination?  e. Is a 500 mL aliquot (or small aliquot diluted to 500 mL if necessary) containing not more than 100 mg/L of cyanide taken for distillation?  f. Is sodium hydroxide used as the absorbing solution?  g. Is a fritted glass disc used to disperse HCN in absorbing solution?  h. Is the vacuum adjusted so that about two air bubbles per second enter the boiling flask?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
i. Is lead acetate paper used to check the sample for the presence of sulfide?		
j. If the test is positive, is bismuth nitrate solution used to remove sulfide?		
k. If samples are known or suspected to contain nitrate/nitrite, is adequate amount of sulfamic acid solution added, after the air rate is set, to remove nitrate/nitrite?		
l. Are sulfuric acid and magnesium chloride added, with washing, through the air inlet tube?		
m. Is the sample heated to boiling and then refluxed for one hour?		
n. After the reflux period is completed, is heat turned off and the airflow continued for at least 15 minutes?		
o. Are the contents of the gas absorber drained into a 250 mL volumetric flask?		
p. Are the gas absorber and the tube connecting the reflux condenser with the gas absorber rinsed with distilled water and combined with the drained liquid in the volumetric flask and the contents diluted to 250 mL?		
q. If incomplete recovery is suspected, is a fresh charge of sodium hydroxide placed in the gas washer and the sample refluxed for one more hour?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
r. If samples contain appreciable amount of solid, oil, or grease to interfere with homogenization and agitation of the sample mixture in the distillation flask, is Method 9013 used to extract cyanide?  s. Is Method 9013 used for the extraction of soluble cyanides from oil, solid, and multiphase samples?		
Sample Analysis:  a. Is the amount of sodium hydroxide in the standards and the samples analyzed the same?  b. Is the chlorine demand of any compounds in the distillate tested with KI-starch paper?  c. Do standards bracket the concentration of the samples?  d. If dilution is required, is distillate diluted with method blank solution?  e. If pyridine-bartituristic acid is used, are the reagents mixed and the color allowed to develop for 8 minutes before reading being taken within 15 minutes?		
Quality Control:  a. Are all QC data maintained and available for easy reference and inspection?  b. Is a three-level data review carried out within the lab prior to data release?		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
c. Is a lab specific MDL empirically established and updated on a semiannually basis?		
d. Does the lab specific MDL meet or exceed the method specified MDL?		
e. Is a method blank run at a minimum rate of 5% or one per batch, whichever is more frequent?		
f. Is calibration curve verified within $\pm 15\%$ of an independent, mid-range check standard for each batch?		
g. Is a matrix spike sample at a level of 40 $\mu\text{g/L}$ analyzed per batch to check the efficiency of distillation?		
h. Are duplicate analyses performed at a minimum rate of 5% or one per batch, whichever is more frequent?		
i. Is one pair of matrix spike and matrix spike duplicate samples run at a minimum rate of 5% or one per batch, whichever is more frequent?		
j. Is method of standard additions used for the analysis of all samples that suffer from matrix interferences?		
k. Are control charts for internal QC data plotted and available to bench chemists?		
l. Are control limits for internal quality control empirically established and updated on a regular basis?		

**CHART I-31**

**CLASSICAL ANALYSIS: CYANIDE (9010A)**

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ITEM	YES	COMMENT
<p>Data Package:</p> <p>a. Does the length of storage time for all sample related information, including chain-of-custody, instrument calibration, sample preparation and analysis, etc., comply with regulatory requirements, organizational policy, or project requirements, whichever is more stringent? (It is recommended that documentation be stored for a minimum of three years from submission of the project final report.)</p> <p>b. Does the data package contain all method required QC data and meet the USACE contract requirements?</p> <p>c. Are all raw data signed and dated by the persons who performed the sample analysis and data review?</p>		
<p>Waste Disposal:</p> <p>a. Does the lab use a contractor to dispose of residual and prepared samples, and samples with analysis cancelled?</p> <p>b. Are lab wastes disposed of properly such that no secondary pollution is produced by sample analysis and the USACE will not be liable for any pollution problems in the future?</p>		
<p>Overall Evaluation:</p> <p>a. Does the lab have sound technical capability for cyanide analysis?</p>		

CHART I-31

CLASSICAL ANALYSIS: CYANIDE (9010A)

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ITEM	YES	COMMENT
b. Does the lab have appropriate capacity to handle the contract load? Average number of samples analyzed and reported per month: _____		
c. Could the lab handle quick turnaround samples?		
d. Overall, is the lab acceptable for cyanide analysis?		
Additional observations, comments, or problems:		

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**CHART I-32**

**CLASSICAL ANALYSIS: TOC (9060)**

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ITEM	YES	COMMENT
Are samples preserved on collection by adjusting the pH to two or less with sulfuric acid or hydrochloric acid and cooling to 4°C?		
Are samples analyzed within 28 days of collection?		
Based on the preliminary treatment of the samples prior to analysis, is a notation made defining the type of carbon to analysis?		
Is carbon dioxide-free double distilled water used on the preparation of standards and dilution of samples?		
Is the use of ion exchanged water avoided?		
Is the potassium hydrogen phthalate stock solution prepared using primary standard grade reagent?		
Is the hypodermic needle size selected so as to obtain the most reproducible results?		
Are injections repeated until three consecutive peaks are obtained that are reproducible to within $\pm 3\%$ ?		
Does the series of standards run encompass the expected concentration range of the samples to be run?		
Is a dilution water blank run?		
Quality Control Requirements:  a. Is a laboratory blank analyzed daily or with each sample run?		

CHART I-32

CLASSICAL ANALYSIS: TOC (9060)

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ITEM	YES	COMMENT
b. Is a reference standard analyzed with every tenth sample?		
c. Is a spiked sample analyzed with every 20th sample?		
d. Are duplicate analyses performed on a minimum of 10% of all positive samples?		
Additional observations, comments, or problems:		

CHART I-33

WASTE CHARACTERISTICS: IGNITABILITY (1010/1020)

Page 1 of 3

ITEM	YES	COMMENT
Pensky-Martins Closed Method (1010):  a. Is the Pensky-Martins closed-cup method used to determine the flash point of liquids that tend to form surface films under test conditions or contain non-filterable suspended solids?  b. Are two standard thermometers available?  c. Is a barometer capable of measuring ambient pressure available (barometers precorrected to give sea-level reading are not acceptable)?  d. Are results documented with the following information?  (1) Observed flash point?  (2) Ambient barometric pressure?  (3) Corrected flash point?  e. Is a duplicate sample included with every tenth sample?  f. Is a p-xylene reference standard determined in duplicate with every sample batch?  g. Is the average of the duplicate p-xylene reference standard flash point determination $27 \pm 0.8^{\circ}\text{C}$ ( $81 \pm 1.5^{\circ}\text{F}$ )?		

CHART I-33

WASTE CHARACTERISTICS: IGNITABILITY (1010/1020)

Page 2 of 3

ITEM	YES	COMMENT
<p>Setaflash Closed-Cup Method (1020):</p> <p>a. Is the Setaflash closed-cup method used to determine the flash point of liquids that have flash points between 0° and 110°C (32° and 230°F) and viscosities lower than 150 stokes at 25°C (77°F)?</p> <p>b. Are ASTM grade thermometers available?</p> <p>c. Is heat transfer paste available?</p> <p>d. Is a barometer capable of measuring ambient pressure available (barometers precorrected to give sea-level reading are not acceptable)?</p> <p>e. Are results documented with the following information?</p> <p>(1) Observed flash point?</p> <p>(2) Ambient barometric pressure?</p> <p>(3) Corrected flash point?</p> <p>f. Is a duplicate sample included with every tenth sample?</p> <p>g. Is a p-xylene reference standard determined in duplicate with every sample batch?</p> <p>h. Is the average of the duplicate p-xylene reference standard flash point determination <math>27 \pm 0.8^{\circ}\text{C}</math> (<math>81 \pm 1.5^{\circ}\text{F}</math>)?</p>		

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CHART I-33

**WASTE CHARACTERISTICS: IGNITABILITY (1010/1020)**

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ITEM
Additional observations, comments, or problems:

CHART I-34

WASTE CHARACTERISTICS: CORROSIVITY (1110)

Page 1 of 1

ITEM	YES	COMMENT
Are the steel coupons of SAE Type 1020 steel?		
Are the steel coupons suspended and supported with a non-conducting material such as glass, fluorocarbon, or coated metal?		
Are the areas of coupons known to ±1%?		
Is a blank run with each test sample?		
Are duplicates run with every tenth sample?		
Is cleaning done by either mechanical, chemical, or electrolytic means?		
Additional observations, comments, or problems:		

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**CHART I-35**

**WASTE CHARACTERISTICS : Toxicity (1311)**

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ITEM	YES	COMMENT
Is reactivity determined by the written criteria and an impact apparatus based on an 8-lb weight?		
Total Reactive Cyanide:  a. Is the approved test method (Method 9010A) in place?  b. Are the samples collected with minimum aeration and headspace, kept in a cool dark place, and analyzed as soon as possible?  c. Equipment: Are the following pieces of equipment available?  (1) Three neck round bottom flask of 500-mL capacity?  (2) Separator funnel with pressure equalizing tube and 24/80 ground glass joint and Teflon sleeve?  (3) Water pumped or oil pumped nitrogen gas?  (4) Rotometer?  d. Method Verification:  (1) Has the system been checked with a reference solution yielding a recovery greater than 50%?  (2) Has this been documented?		
Total Reactive Sulfides:  a. Is the approved test method (Method 9030) in place?		

CHART I-35

WASTE CHARACTERISTICS: REACTIVITY (SECTION 7.3)

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ITEM	YES	COMMENT
b. Are the samples collected with minimum aeration and headspace, kept in a cool dark place, and analyzed as soon as possible?		
c. Equipment:  (1) Is the apparatus required for Method 9030 available?		
(2) Has the absorber been replaced with an "Industrial Hygiene" type detection tube for sulfide (100 to 2,000 ppm)?		
d. Method Verification:  (1) Has the system been checked with a reference solution yielding a recovery greater than 50%?		
(2) Has this been documented?		
Additional observations, comments, or problems:		